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Laboratory Methods for Testing Peat—Ontario Peatland Inventory Project

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by J.L. Riley

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Foreword

The Ontario Peatland Inventory Project was a component of the Hydrocarbon Energy Resources Program (HERP) of the Ontario Geological Survey. This inventory of the peat and peatland resources of Ontario was intended to provide information on possible fuel peat deposits as a means of encouraging increased energy self-sufficiency in Ontario. It also provided information on the resources of horticultural peat in the province. In addition, data on the distribution, frequency, and types of peatlands in Ontario assists in land use planning and disposition.

To complement the previously released Open File Reports on specific peatland study areas across the province, the inventory undertook laboratory analyses of over 1700 peat samples collected during the course of studies. The analysis of these samples entailed the development and standardization of required laboratory methods, and the assessment of the precision and accuracy of the reported data.

This report details the specific laboratory methods which were used, and evaluates the results of internal and blind quality control tests. It also reports the results of several tests of particular methods, and suggests the precision levels which may be anticipated from the use of these techniques. The final analytical results of these tests have been released in Open File Reports on different regions of the province.

V.G. Milne

Director
Ontario Geological Survey

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CONVERSION FACTORS FOR MEASUREMENTS IN ONTARIO GEOLOGICAL SURVEY PUBLICATIONS

Conversion from SI to Imperial			Conversion from Imperial to SI		
SI Unit	*Multiplied by*	*Gives*	*Imperial Unit*	*Multiplied by*	*Gives*
		LENGTH			
1 mm	0.039 37	inches	1 inch	**25.4**	mm
1 cm	0.393 70	inches	1 inch	**2.54**	cm
1 m	3.280 84	feet	1 foot	**0.304 8**	m
1 m	0.049 709 7	chains	1 chain	20.116 8	m
1 km	0.621 371	miles (statute)	1 mile (statute)	**1.609 344**	km
		AREA			
1 cm^2	0.155 0	square inches	1 square inch	**6.451 6**	cm^2
1 m^2	10.763 9	square feet	1 square foot	**0.092 903 04**	m^2
1 km^2	0.386 10	square miles	1 square mile	2.589 988	km^2
1 ha	2.471 054	acres	1 acre	0.404 685 6	ha
		VOLUME			
1 cm^3	0.061 02	cubic inches	1 cubic inch	**16.387 064**	cm^3
1 m^3	35.314 7	cubic feet	1 cubic foot	0.028 316 85	m^3
1 m^3	1.308 0	cubic yards	1 cubic yard	0.764 555	m^3
		CAPACITY			
1 L	1.759 755	pints	1 pint	0.568 261	L
1 L	0.879 877	quarts	1 quart	1.136 522	L
1 L	0.219 969	gallons	1 gallon	**4.546 090**	L
		MASS			
1 g	0.035 273 96	ounces (avdp)	1 ounce (avdp)	28.349 523	g
1 g	0.032 150 75	ounces (troy)	1 ounce (troy)	**31.103 476 8**	g
1 kg	2.204 62	pounds (avdp)	1 pound (avdp)	**0.453 592 37**	kg
1 kg	0.001 102 3	tons (short)	1 ton (short)	**907.184 74**	kg
1 t	1.102 311	tons (short)	1 ton (short)	**0.907 184 74**	t
1 kg	0.000 984 21	tons (long)	1 ton (long)	**1016.046 908 8**	kg
1 t	0.984 206 5	tons (long)	1 ton (long)	**1.016 046 908 8**	t
		CONCENTRATION			
1 g/t	0.029 166 6	ounce (troy)/ ton (short)	1 ounce (troy)/ ton (short)	34.285 714 2	g/t
1 g/t	0.583 333 33	pennyweights/ ton (short)	1 pennyweight/ ton (short)	1.714 285 7	g/t

OTHER USEFUL CONVERSION FACTORS

1 ounce (troy) per ton (short)	20.0	pennyweights per ton (short)
1 pennyweight per ton (short)	0.05	ounces (troy) per ton (short)

Note: Conversion factors which are in bold type are exact. The conversion factors have been taken from or have been derived from factors given in the Metric Practice Guide for the Canadian Mining and Metallurgical Industries, published by the Mining Association of Canada in cooperation with the Coal Association of Canada.

Laboratory Methods for Testing Peat—Ontario Peatland Inventory Project

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Abstract

Between 1983 and 1985, peat samples were analyzed from 154 peatlands across Ontario south of 51°N latitude. The number of individual samples was 1733, taken from 341 peat cores. The analyses were undertaken by a commercial laboratory, to specifications for methodology developed for the Ontario Peatland Inventory Project.

The analyses included parameters relevant to both the energy and horticultural peat characteristics of the materials. These included peat pH (H_2O and $CaCl_2$), cation exchange capacity (meq/100g, corrected to dry weight), conductivity (μmhos/cm at 25°C), fiber content (mechanical dispersion method, percent, dry weight), moisture content (percent, wet weight), bulk density (g/cm^3, dry and wet), absorptive capacity, ash content (percent, dry weight), volatile matter (percent, dry weight), net calorific value (cal/g, dry weight, corrected for N, H, S), C (total and organic, percent, dry weight), N, H, S (all percent, dry weight), O (by difference), Ca, Fe, Al, Mg, Mn, P, K, Pb, Cu, Zn, Hg, As (all ppm, dry weight).

The methods used in this project are documented here to clarify site data published by the Ontario Peatland Inventory Project, and to allow other similar projects to compare laboratory results with this data set. The results of blind duplicate and blind replicate tests, and standards and blanks, are presented as documentation of the accuracy and precision of the results, and the "practical determination levels" achieved by individual methods.

Several tests of methods were undertaken to investigate modifications of recommended methods, and to clarify the relationship between results by the methods indicated and by other commonly used techniques to measure the same parameters. Tests were made of certain aspects of the following methods: cation exchange capacity, conductivity, pH, fiber content, rehydration of dried, homogenized peats, ash content, and various wet-ashing and dry-ashing dissolution techniques preparatory to multielement ICAP spectrometry.

Résumé

Des échantillons de tourbe provenant de 154 tourbières de l'Ontario situées au sud du 51° de latitude nord ont été analysés entre 1983 et 1985. On a prélevé 1733 échantillons individuels de 341 carottes de tourbe. Les analyses ont été effectuées par un laboratoire commercial, conformément à la méthodologie établie pour le projet d'inventaire des tourbières en Ontario (Ontario Peatland Inventory Project).

Les analyses portaient sur les caractéristiques énergétiques et horticoles des échantillons, notamment sur le pH de la tourbe (H_2O et $CaCl_2$), le pouvoir d'échange cationique (meq/100g, rectifié au poids sec), la conductivité (µmhos/cm à 25°C), la teneur en fibre brute (méthode de la dispersion, pourcentage, poids sec), la teneur en eau (pourcentage, poids frais), la masse volumique en vrac (g/cm^3, sec et frais), la capacité d'absorption, la teneur en cendre (pourcentage, poids sec), la matière volatile (pourcentage, poids sec), le pouvoir calorifique inférieur (cal/g, poids sec, rectifié pour N, H, S), la teneur en C (total et organique, pourcentage, poids sec), N, H, S (tous les pourcentages, poids sec), O (par différence), Ca, Fe, Al, Mg, Mn, P, K, Pb, Cu, Zn, Hg, As (tous les ppm, poids sec).

Les méthodes utilisées ont été décrites afin d'éclairer les données sur les emplacements publiées par le projet d'inventaire des tourbières en Ontario et de permettre aux projets analogues de comparer leurs résultats en laboratoire avec cet ensemble de données. Les résultats des essais aveugles en double, des essais aveugles répétés, des essais de référence et des essais à blanc servent à illustrer l'exactitude et la précision des résultats, et les "niveaux de détermination pratiques" atteints par l'entremise de chaque méthode.

On a soumis les méthodes à des essais afin d'étudier les modifications recommandées, et de mieux comprendre le lien qui existe entre les résultats obtenus à l'aide des méthodes indiquées et ceux obtenus à l'aide des techniques traditionnellement utilisées pour mesurer ces paramètres. Les essais portaient sur certains aspects des méthodes suivantes, soit le pouvoir d'échange cationique, la conductivité, le pH, la teneur en fibre brute, la réhydratation de la tourbe sèche ou homogénéisée, la teneur en cendre, et sur diverses techniques de dissolution par voies humide et sèche des cendres préalables à la spectrométrie ICAP multiéléments.

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Introduction

In 1981, the Hydrocarbon Energy Resources Program of the Ontario Geological Survey initiated a project to investigate the potential of peat resources in Ontario. The report entitled "Evaluation of the Potential of Peat in Ontario" (Monenco Ontario Limited 1981) recommended a provincial inventory as the means of quantifying and documenting this potential. As a result, a Peatland Inventory Project was initiated, and proceeded with investigations of peatlands in northwestern Ontario (Riley and Michaud 1989), northeastern Ontario (Riley 1986), and southeastern Ontario (Riley 1988).

Prospective peatlands were selected on the basis of size, peatland type, proximity to population centres, and accessibility. On each detailed study site, a sampling grid was designed, made up of baselines and intersecting sidelines at 500 m intervals. Points at each 100 m along these transects were described in terms of surface vegetation, site wetness, and elevation, the latter either by transit, electronic topochain, photogrammetry or airborne laser profiling. At each sample point, a core was pulled and characterized stratigraphically by change in peat material, fiber content, and wetness (Riley 1984b).

For laboratory analyses, field workers recovered top-to-bottom sample cores from representative points on peatlands, following field studies at the site. Up to four sample cores were recovered for laboratory analysis, from points chosen to reflect the general patterns of peat stratigraphy, peat depths, and peatland vegetation.

From 1983 to 1985, laboratory analysis was undertaken of peat cores representative of the peat materials of 154 peatlands studied in detail across Ontario. The number of cores analyzed was 56 in 1983, 169 in 1984, and 116 in 1985. Each core was subsampled in the field with respect to changes in peat humification (von Post scale) and/or perceived peat type (botanical constituents). As a result, the total number of peat samples was 1733, with numerous additional control samples as well. These cores were retrieved with either a Mini-Macaulay auger (5 cm diameter chamber) (National Research Council of Canada (NRCC) 1979), or a modified piston sampler (4 cm diameter chamber) (Korpijaakko 1981).

The laboratory analysis was designed to complement the detailed site data published on each deposit, so that laboratory data on each core could be stratigraphically integrated with transect profiles of peat types and degrees of humification, and with other peatland data. The detailed studies also included elevation contouring, depth contour mapping, volume calculations, peatland classification mapping, and summary text. As a result, the laboratory test data is viewed as critical data in documenting the resource potential of individual sites.

The laboratory tests were undertaken to reflect the broad objectives of the Peatland Inventory Project. Both energy and horticultural peat resources were considered in the inventory, and the laboratory methods relate to both of these potential resource uses. In addition, the test data may also relate to agricultural potential of some sites, as well as other future uses.

All tests were undertaken on samples taken from the field and preserved in a frozen state until analysis. As a result, some measurements may not equate with similar tests done in the field, such as bulk density, which can be measured *in situ*. In some studies, physical tests are conducted on peat materials which have been partially dried or prepared for other purposes; the tests reported here were conducted on peats at or near their field moisture content, in a relatively undisturbed condition. However, these conditions are indicated in the methods, so that appropriate comparison of these test results with those of other data sets may be made.

In designing a methodology to standardize the laboratory tests over the course of the inventory, several major sources were reviewed. Most relevant of these were the Peat Testing Manual (NRCC 1979), the Manual on Soil Sampling Analysis (Black *et al.* 1965), the System of Soil Classification for Canada (Canada Department of Agriculture 1974, p.141-165), the Annual Book of ASTM Standards (American Society for Testing and Materials (ASTM) 1981 and later), Lynn *et al.* (1974), and Walmsley (1977). During the course of analysis, several other relevant reviews appeared; Testing of Peat and Organic Soils (Jarrett 1983) and the Methods of the Finnish Domestic Fuel Laboratory (Sheppard 1984). The methods used in other peat inventories were also reviewed; for example, in Ontario (Graham 1979) and Minnesota (Minnesota Department of Natural Resources (MDNR) 1982).

The analysis of major elements was considered basic to the characterization of the peat materials (NRCC 1979). The occurrence and distribution of these and other elements was considered important in terms of substrate fertility and current site productivity (Stanek and Jeglum 1977), biochemical partitioning of elements (Damman 1978; Pakarinen and Gorham 1983; Kilham 1982), and potential post-combustion pollutants (Glooschenko and Capobianco 1982; Pakarinen *et al.* 1980). Elemental analyses done on all samples included C (total and organic), N, H, S, O (by difference), Ca, P, K, Mn, Mg, Al, Fe, Cu, Zn, Pb, Hg, and As.

Except for Hg, As, C, H, N, and S (and Pb and Cu in 1985), the elemental analysis was done by inductively coupled argon plasma emission spectrometry (ICAP) in order to achieve simultaneous multielement analysis with reasonable sensitivity and low cost (*see* Bratter *et al.* 1983). A review of sample ashing methods (Arafat and Glooschenko 1981; Glooschenko *et al.* 1980) suggested wet-ashing techniques as most suitable. This was reviewed during the course of the analyses, and a more successful dry-ashing method was used during the last year's analysis (*see* Section 3.7.2.).

Results of some elemental tests also related to potential energy production, both in terms of energy output, design of boilers, and possible requirements for control of emissions; for example, C, H, N, S, and others.

Other energy-related tests included the heating value, volatile matter, and ash content of the peat. These tests, and all elemental analyses, are reported on a dry basis (0 percent moisture content). In many published methodologies, results on a dry basis are calculated by performing independent, parallel moisture determinations, with each result then recalculated to a dry basis. This technique was investigated and found to be very time consuming in the course of a large run of samples. As an alternative, all field samples were dried slowly at 50°C for 1 to 4 weeks, then ground and homogenized, further dried at 60° to 80°C for 16 hours, and bottled. They were then re-dried to 0 percent moisture content at 90°C immediately before dry tests were done. Because of the number of samples involved, air drying was not possible in this project.

Tests were undertaken of the problem of sample rehydration in laboratory conditions (Section 3.5), and measures taken to ensure that all dry tests were done on samples at 0 percent moisture content. It was recognized that this, and perhaps any, drying method, entails some mild oxidation, particularly of mercury. For this element, subsamples of the wet peat were dried separately at 50°C. In 1985 analyses, dry ashing was used in preparing samples for ICAP multielement spectrometry. Ashing at a "medium temperature" (400°C) was conducted for this purpose in order to avoid decompositional problems associated with higher temperatures (Andrejko *et al.* 1983).

Calorific values (in cal/g) are all expressed as net calorific values on a dry basis, corrected for H, N, and S determined independently. In this regard it should be noted that while much of the Fennoscandian literature and all payments for commercial energy peat producers in Finland are based on net calorific values, much of the North American literature reports the higher gross calorific values, perhaps because the United States Department of Energy defines fuel grade peat in terms of gross values. Conversion factors are presented in Section 2.13, and care must be exercised in comparing results from different laboratories.

The ash content of organic materials is important both in terms of definition of peat materials (less than 25 percent ash; Andrejko *et al.* 1983) and combustion systems for fuel peats (Sheppard 1984). A test was made of ashing regimes at different temperatures over different periods of time (Section 3.6). This test confirmed the recommendation of 750°C by Andrejko *et al.* (1983). After further tests, a one hour time period, with a mid-point removal of sample for five minutes to replenish the oxygen supply, was considered satisfactory if extreme care was taken to check that complete ashing, without unburned particles or charring, was accomplished. This is an attractive duration for large commercial sample runs, but it is recommended that similar corroborative tests be undertaken by any laboratory assessing the ash content of peat.

Other tests to evaluate the nutrient capabilities of the peat samples were cation exchange capacity (CEC), pH (H_2O and $CaCl_2$), and conductivity.

Tests to characterize physical aspects of the peat relevant to materials handling, horticultural potential (Farnham 1968), and peatland hydrology (Boelter 1968) included moisture content, absorptive capacity, bulk density, and fiber content. These measurements were made on samples taken directly from the field, so that their direct comparability with similar measurements made either *in situ* or after site development or peat harvesting, is limited. However, these methods are defined in sufficient detail to allow the development of conversion factors where necessary.

One example of the need for such conversion factors occurs with fiber content. There are a number of different recommended methods (Levesque and Dinel 1977; McDonnell and Farrell 1984), some of which are highly reproducible only after considerable experience with different peat materials (e.g., "syringe method"). In order to standardize a particular method at a reasonable level of sensitivity, a mechanical dispersion method was adopted, and through the co-operation of Agriculture Canada, the correlation between the results of this method and the results of the syringe method's unrubbed fiber determination was evaluated. As a result, the fiber content values produced by the method outlined in this report can be correlated with those of data sets based on other methods.

The review of published methods of peat analyses showed an apparent weakness in terms of both tests of methods and the assessment of the precision and accuracy which may be expected from the application of such methods. This affects the comparability of results between various laboratories, and contributes to a lack of background data available to laboratories for setting their own internal quality control standards. This is compounded by the absence of peat "reference materials", such as the

United States National Bureau of Standards materials. While it was not the mandate of this Inventory Project to address these problems, several by-products of this analytical program may be of use to others, and may encourage other workers to improve both analytical methods and standards of precision and accuracy.

Firstly, a few tests of methods were attempted in order to clarify the methodology (Section 3.0). These included a test of: 1) nitrogen (N) levels in the alcohol washes and in blank runs of the cation exchange capacity procedure; 2) the effect of adding various amounts of water to peat samples on the measured peat pH; 3) the effect on conductivity of adding increasing amounts of water to insufficiently wetted samples; 4) a mechanical dispersion method of fiber content determination; 5) rehydration effects on homogenized oven-dry peat; 6) different ashing regimes; and 7) wet-ashing and dry-ashing dissolution methods preparatory to ICAP spectrometry.

Secondly, several "test peats" were used in the course of analyses as bulk samples incorporated as blind replicates throughout the sample runs. Such peats have considerable value in assessing the precision of various methods involving wet and dry tests, especially if the test peat is near the lower limit of the range of analyte values for the peat samples as a whole. In this regard, it is unlikely that any "reference material" useful for wet tests will ever be practical due to the difficulties of homogenizing, storing, shipping, and maintaining saturated peats. Another peat material was developed as an internal laboratory standard (Geoscience Laboratories, Ontario Geological Survey, Toronto), and was subjected to a complete elemental analysis by two independent "wet chemistry" methods per element. This dried, homogenized peat standard provided considerable control on accuracy of tests undertaken by other laboratories using other techniques of elemental analysis (*see* Section 3.7.2).

About 10 percent of all the samples were two independent blind replicates in both 1984 and 1985, and about 5 percent of samples were blind duplicates. The results of these control samples were analyzed in order to assess the precision of the results, based on a requirement of 95 percent confidence levels and based on "practical determination levels" (PDLs) required of each of the methods. Equipment detection limits such as those normally made available by laboratories were judged to be of only secondary interest because our primary interest was in maximizing the precision of complex tests on relatively complex organic materials. An analysis was made of these results (Section 4.0) in order to evaluate the "effective" practical determination levels achieved by these methods during the course of this project, and to extrapolate to the practical determination levels required of each method to render 80 percent of the blind duplicate pairs acceptable. These results (*see* Tables 4a through 4d) suggest the scale of the determination limits which may be expected of these methods in the course of a very large run of samples. Their precision and accuracy were judged to be acceptable to the Inventory Project, but may well be improved upon in other projects using different laboratory methods.

The analytical procedures presented here were developed as project specifications for tendering contracts for peat analyses to commercial laboratories, at which time the laboratories were also informed of the precision and accuracy requirements of the project, including required determination limits for each test. In each of the three years of analyses, Technical Service Laboratories (Mississauga) conducted all of the laboratory work, except where data on standards, controls, and tests are indicated to be from other sources.

The peat data itself and regional analyses of peat stratigraphy based on the laboratory data has been published in reports on specific peatlands of the province (Riley 1986, 1988; Riley and Michaud 1989). It is the intention of this report to provide technical detail on the analytical methodology of the laboratory aspects of the Peatland Inventory Project as background to the reports on the overall inventory results.

This report intends no implicit endorsement of these particular techniques or equipment. Numerous alternative methods are available to laboratories for similar analyses, and the capabilities of particular laboratories greatly influence the regimen of testing. Documentation of these methods should, however, enable other workers to clarify the results of other tests in comparison to these.

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1.0 Collection and Handling of Peat Samples

1.1 COLLECTION OF SAMPLES

At representative points on peatlands investigated in detail, complete cores were recovered from the surface of the peat down to the underlying mineral substrate. These sampling points were selected to represent the variation in peat stratigraphy and/or vegetation occurring on the peatland. Mini-Macaulay samplers were recommended for this work (5 cm diameter, 50 cm long).

The cores were divided into intervals on the basis of changes in botanical composition and degree of humification (von Post scale) (Henderson and Doiron 1982). The minimum sample weight of each core interval collected was 1 kg and detailed core logs were recorded at each sampling point. Sedimentary peats (ooze) and marl intervals were also sampled by the same procedure.

Samples of each core interval were sealed in plastic bags with excess air expelled. These samples were double bagged, sealed to prevent water loss, and clearly labelled on the outside as to project area (e.g., Ignace), peatland number (e.g., 52G/191), sample point number (e.g., L3200N+200E), and stratum level (e.g., C2). The samples from all intervals of a particular core were then bagged together, with another outside label as to project area, peatland number, and sampling point number. The samples were stored in the dark.

Physical samples were frozen as soon as possible in the field or kept at temperatures less than 3°C (Jasieniuk and Johnson 1982; Levesque *et al.*

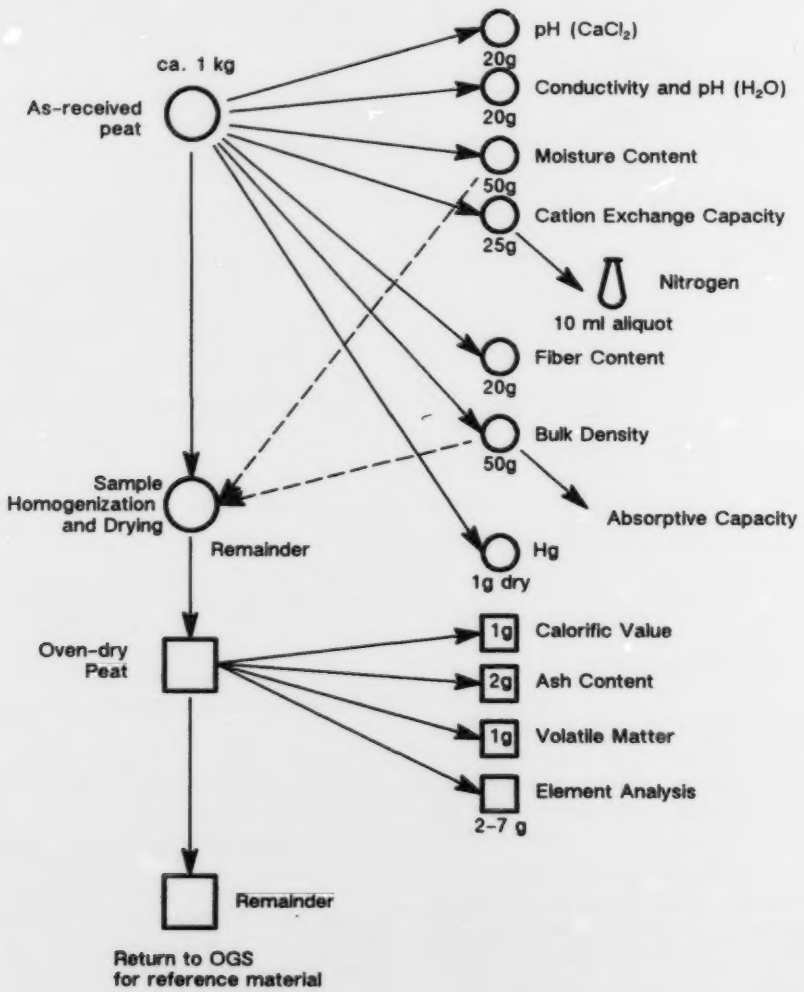


Figure 1. *General laboratory handling of peat samples.*

1980). All samples were frozen when they were brought from the field.

Before delivery to the laboratory, selected specimens were briefly thawed in order to allow blind duplicates to be made, and other samples to be added as blind (disguised) replicates. They were delivered to the laboratory frozen.

1.2 GENERAL LABORATORY HANDLING OF WET PEAT SAMPLES

The general laboratory handling of peat samples is illustrated in Figure 1.

All of the subsamples for wet peat tests were separated and weighed after opening and mixing of the wet, thawed peat.

The peat bag was opened and dumped on a plastic sheet, and thoroughly mixed by hand.

A portion was removed and placed in a high wet strength kraft bag for mercury analysis (Section 2.22). The sample was dried at 50°C in the bag.

Wet mixed peat was weighed into numbered paper cups for pH (H_2O) and conductivity, and pH ($CaCl_2$) measurements (Sections 2.2, 2.3, and 2.4).

A weighed portion was placed in a pre-weighed and numbered aluminum dish for moisture determination (Section 2.6).

Weighed portions were placed in two 300 ml plasticized paper tubs for cation exchange capacity measurement (Section 2.1) and for fiber content measurement (Section 2.5).

A weighed portion was placed in a glass beaker for determination of bulk density and absorptive capacity (Sections 2.8 and 2.9).

The remainder (approximately 1 kg) was placed in a large aluminum tray for drying prior to homogenization (Section 2.10) for subsequent dry tests.

2.0 Analytical Methodology

2.1 CATION EXCHANGE CAPACITY (AS-RECEIVED PEAT)

Cation exchange capacity (CEC) is the total amount of exchangeable cations that can be held by a peat (NRCC 1979; McKeague 1976). Horticultural peats should have a cation exchange capacity of 90 meq/100g or greater (Farnham 1968).

The method is taken from the NRCC (1979, p.113, "CEC (of Fresh Sample) by NH_4OAc at pH 7.0"), corrected to dry peat weight on the basis of moisture content as calculated in Section 2.6. Sample: as-received, 25 g. Result: cation exchange capacity, meq/100g, corrected to oven-dry weight.

2.1.1 REAGENTS FOR EXTRACTION

Ammonium acetate (pH 7), NH_4OAc, 1M. Measure 300 ml deionized water into a 1 l graduated cylinder. Add 575 ml glacial acetic acid and 600 ml of 29 percent NH_4OH solution and mix. Dilute to 10 l and mix thoroughly. Check that pH is 7.00 ± 0.05 and adjust with drops of acetic acid or ammonia as necessary.

Isopropyl alcohol, 60 percent by volume.

Potassium chloride, KCl 5 percent wt/volume.

2.1.2 PROCEDURES

2.1.2.1 Extraction Using Neutral Ammonium Acetate

Add 200 ml 1M NH_4OAc extractant to 25 g of fresh mixed peat in a plastic lined tub, stir, and leave 10 minutes.

Pour liquid and peat sample into a 14 cm Buchner funnel containing No.44 filter paper and filter into a dry filter flask.

Remove excess extractant by washing residue 4 times with 50 ml portions of isopropyl alcohol. Reject all washings.

Rinse flask with alcohol, then rinse with water.

2.1.2.2 Leaching with Potassium Chloride Solution

Leach the residue on the filter with 30 to 50 ml portions of KCl solution to displace adsorbed NH_4^+–Nitrogen. Allow time for filtering between additions and continue until almost 250 ml has been collected. Top to 250 ml with deionized water.

2.1.2.3 Determination of the NH_4^+–Nitrogen Acid

Transfer a 25 ml aliquot into an Automatic 16200 Kjel-Foss Nitrogen Apparatus and determine the nitrogen content. Calibrate the Kjel-Foss Apparatus using $(NH_4)_2SO_4$ standards at approximately 0.015 percent and 0.3 percent nitrogen.

2.1.2.4 Calculation

Calculate the cation exchange capacity, in meq/100g, as follows:

$$CEC = \frac{\text{weight of nitrogen in bottle (mg)}}{\dfrac{14.0067}{\text{sample dry weight (g)}}} \times 100$$

$$= \frac{\%N \times V}{(100 - \%M) \times W} \times \frac{10^5}{14.0067}$$

where $\%N$ = percent nitrogen in aliquot,
V = volume of solution in bottle, in millilitres,
$\%M$ = percent moisture, determined in Section 2.6,
W = weight of sample as received, in grams.

In these analyses, the formula became:

$$CEC = \frac{\%N \times 71394.4}{100 - \%M}$$

2.2 pH in H_2O

Measures hydrogen ion concentration (after NRCC 1979; MDNR 1982; Levesque *et al.* 1980; ASTM D2976-71).

Sample: as-received, 20 g. Result: pH to nearest 0.1 pH unit.

2.2.1 PROCEDURE

Weigh 20.0 g of thoroughly mixed peat into a paper cup.

Add 20 ml of deionized H_2O (pH 6.6 to 7.5, boiled at least 1 hour to ensure it is free of CO_2). For fibrous samples which are not sufficiently wetted for pH and conductivity measurement, add 80 ml of distilled H_2O.

(To ensure CO_2-free deionized water, the boiled distilled water is added using a suitable dispenser drawing from a reservoir equipped with KOH traps on the replacement air source.)

Cover and shake on a reciprocating shaker for 10 minutes.

After shaking allow to sit for 30 minutes in the sealed container.

Measure the pH on a pH meter accurate to 0.1 units, equipped with temperature compensation. Standardize the pH meter routinely using buffers of known pH (Potassium Acid Phthalate, pH 4.01±0.01; Phosphate Buffer Solution, pH 7.00±0.01).

NOTE: If measuring both pH and conductivity on the same portion, the conductivity should be measured first.

2.3 pH in $CaCl_2$

Measures hydrogen ion concentration independent of initial amounts of salts present.

Sample: as-received, 20 g. Result: pH to nearest 0.1 pH unit.

2.3.1 PROCEDURE

Weigh 20 g of thoroughly mixed fresh peat into a paper cup.

Add 20 ml of 0.01 M $CaCl_2$ solution. Add 80 ml for fibrous samples which required 80 ml added in Section 2.2.

Cover and shake on a reciprocating shaker for 10 minutes.

Allow to sit for 30 minutes covered.

Measure the pH on a pH meter accurate to 0.1 units equipped with temperature compensation. Standardize the pH meter using buffers of known pH as in Section 2.2.1.

2.4 CONDUCTIVITY

Conductivity indicates total concentration of various dissolved ions, excluding hydrogen ions (after NRCC 1979).

Sample: as-received, 20 g. Result: conductivity in µmhos/cm at 25°C.

2.4.1 PROCEDURE

Weigh 20 g of thoroughly mixed fresh peat into a paper cup.

Add 20 ml of deionized H_2O (pH 6.5 to 7.5 boiled free of CO_2). For fibrous samples which are not sufficiently wetted for conductivity and pH measurement, add 80 ml distilled deionized H_2O.

(Boiled distilled water is added using a suitable dispenser drawing from a reservoir equipped with KOH traps on the replacement air source.)

Cover and shake on a reciprocating shaker for 10 minutes.

Allow to sit for 30 minutes after shaking.

Measure the conductivity of the supernatant using a conductivity meter and cell with a constant of 1.0 cm^{-1}.

Standardize the cell routinely against 0.01 N KCl at 25°C (conductivity of 1300 µmhos/cm) or record the temperature and correct the reading according to the following formula:

$$L_{25} = \frac{L_t}{1 + 0.02\Delta t}$$

where L_{25} = conductivity at 25°C,
L_t = conductivity at measured temperature,
Δt = difference between measured temperature and 25°C.

If L_t above 25°C, then Δt is + (positive).
If L_t below 25°C, then Δt is − (negative).

Immediately following the conductivity measurement, the pH may be measured on the same supernatant solution (Section 2.2). The conductivity measurement must be made prior to the pH measurements.

If the pH of the supernatant is 5.1 or lower, the conductivity is corrected by subtracting conductivity owing to hydrogen ions as follows; in which case, the data are reported at L_{25}, H^+ corrected.

pH	H^+ (µmhos/cm at 25°C)
3.0	350.0
3.1	278.0
3.2	220.3
3.3	175.5
3.4	139.3
3.5	110.7
3.6	87.9
3.7	69.8
3.8	55.5
3.9	44.0
4.0	35.0
4.1	27.8
4.2	22.1
4.3	17.5
4.4	13.9
4.5	11.1
4.6	8.8
4.7	7.0
4.8	5.6
4.9	4.4
5.0	3.6
5.1	2.8

For samples to which 80 ml distilled deionized water was added, the conductivity is corrected to 20 ml water by multiplying conductivity at 80 ml by the factor 2.4 (*see* Section 3.3).

2.5 FIBER CONTENT

Fiber content represents a laboratory approximation of the degree of fiber decomposition and water-re-

taining capacity of the peat (after NRCC 1979; Levesque and Dinel 1977; ASTM D2607-69).

Sample: as-received, 20 g. Result: fiber, percent oven-dry peat made up of fibers greater than 0.15 mm.

2.5.1 PROCEDURE

Weigh 20 g of thoroughly mixed fresh peat into a 300 ml plasticized paper tub.

Add 200 ml of deionized water.

Cover the sample and let sit overnight.

Mix the sample with a laboratory stirrer at 240 rpm for 10 minutes. The stirring should not break up much of the peat fiber.

Cover the tub and allow to sit for several hours.

Pour the peat into a 100 mesh (0.15 mm) stainless steel sieve.

Wash the fine particles through the screen with tap water, using a flexible shower attachment set at a flow rate of 5 l/minute.

Midway through washing, cover the screen and immerse in a 2 percent HCl solution to dissolve any carbonates that may be present.

Wash until rinse water is clear (approximately 10 minutes).

Collect the fibers and transfer to an aluminum dish.

Dry the fibers at 90°C to a constant weight (24 hours).

Weigh to 0.01 g.

The fiber content is calculated using the following formula:

Fiber Content (percentage of oven-dry peat made up of fibers >0.15 mm)

$$= D/E \times 100$$

where D = dry weight (g) of fiber residue,
E = dry weight (g) of 20 g of original material as calculated from the moisture content (Section 2.6).

(For correlations with rubbed and unrubbed fiber content by the "syringe method", refer to Section 3.4.)

2.6 MOISTURE CONTENT

Oven drying for moisture determination (after NRCC 1979; MDNR 1982; ASTM D2974-71). Moisture content is used to convert the results of tests on as-received peat to a dry weight basis.

Sample: as-received, 50 g. Result: moisture content, percent wet peat, by weight.

2.6.1 PROCEDURE

Weigh 50 g of thoroughly mixed as-received peat into a tared aluminum dish.

Dry at 90°C for 16 hours. (This temperature was chosen to avoid loss of volatiles. The National Bureau of Standards (NBS) recommends this temperature for drying of all organic standard materials.)

Cool, covered with Saran wrap. Reweigh.

The moisture content is expressed as a percent of total weight.

Moisture content, percent total weight (percentage of total weight represents moisture present in the peat).

$$\text{Total Weight, \%} = ((A-B) \times 100)/A,$$

to nearest 0.1%

where A = grams of as-received sample,
B = grams of oven-dry sample.

Since moisture is used in calculations for several other procedures (cation exchange capacity, fiber content, absorptive value, bulk density, absorptive capacity), **it is recommended that this variable be measured in duplicate.**

2.7 ABSORPTIVE VALUE

Absorptive value is the ratio of the weight of the water originally retained in the peat to the dry weight of the peat.

The absorptive value is derived from the moisture content calculation and relates only to the water retained by the peat in the field condition in which it was collected.

Absorptive value, dry,

$$= \frac{A-B}{B}$$

where A = grams of as-received sample,
B = grams of oven-dry sample.

2.8 BULK DENSITY

Bulk density is the measure of the weight of a given volume; generally increasing with increasing decomposition, moisture content and mineral content, and decreasing with increasing sphagnum content (after MDNR 1982; Graham 1979). Bulk density can be used to convert water, nutrient, and energy characteristics measured on a unit weight basis to a unit volume basis (e.g., Lucas and Rieke 1968).

2.8.1 PROCEDURE

Take a 150 ml beaker. (Determine the weight of water contained in the beaker at the 80 ml mark, placing the beaker at a standard height to minimize parallax in the volume determination.)

Weigh 50 g of thoroughly mixed fresh peat into the beaker.

Add water and stir by hand until all air is expelled.

Bring the volume to the 80 ml mark (determined at the standard height) ensuring that all of the peat is below the water level.

Weigh the resultant beaker, sample, and water.

The bulk density is calculated as follows, where:

A = grams of sample as-received,
B = volume measured as calibrated, cm^3,
C = grams of sample with water (excluding beaker weight),
D = moisture percent wet.

$$\text{Bulk density, g/cm}^3\text{, oven-dried} = \frac{(A(100-D))}{100} \Big/ (B-(C-A))$$

$$\text{Bulk density, g/cm}^3\text{, as-received} = A \Big/ (B-(C-A))$$

(A 150 ml beaker can only be used to measure reliably to within ±5 ml, adding an unnecessary element of variability to these tests. Graduated cylinders are awkward to fill and empty. Containers which can be filled and sealed, with no air inside, or other vessels which can be read to ±0.5 ml are more desirable. As a result, in 1985, a "cocktail shaker" with a sealable spout and a 250 ml capacity was used, which could measure reliably to ±0.5 ml.)

2.9 ABSORPTIVE CAPACITY

Absorptive capacity is the ratio of the weight of potentially retained water to the dry weight of the peat (after Graham 1979, as "Absorptive Value, dry basis"); decreases with increasing humification of the peat. The reported values relate to more or less undisturbed peat; peat harvesting methods may change a peat's absorptive capacity.

The sample is transferred from the bulk density determination or a new 50 g sample must be weighed. Sample: as-received, 50 g.

2.9.1 PROCEDURE

Weigh a coarse bottom sieve containing a pleated filter basket (coffee-style filter was used, enabling easy removal of sample after the test and containment of sample during the procedure).

Transfer the 50 g sample from bulk density procedure to the filter basket contained in the coarse bottom sieve.

Wash all of the peat into the filter using a wash bottle.

Place the sieve into a shallow pan with water such that the peat stays completely saturated for at least 30 minutes. (The basket type filter is important because a flat filter paper will not necessarily contain the sample when it is sitting submerged.)

Lift the sample and sieve from the reservoir and allow it to drain until there is no free water on the peat (allow 45 to 120 minutes).

Measure the weight of the sieve, filter, and sample. (The filter retains a measurable amount of water when wet, in this case in the range of 6.5 g.)

$$\text{Absorptive capacity} = \frac{H-I}{I}$$

where H = grams of saturated sample,
I = grams of oven-dry sample, calculated from the moisture content (Section 2.6).

2.10 SAMPLE HOMOGENIZATION

Sample homogenization involves the preparation of oven-dry sample for subsequent analyses.

Dry all remaining as-received sample and uncontaminated remains of earlier analyses (e.g., moisture content) at approximately 50°C until most moisture has evaporated (samples were left at 50°C for 1 to 4 weeks). Grind sample using a Wiley Mill, passing it through a 80 mesh sieve (0.18 mm). Further dry the ground sample at 60° to 80°C for 16 hours or more, cover, and cool.

Oven-dry samples will rehydrate to 9 to 18 percent moisture at room temperature and humidity (Section 3.5). Therefore, dried homogenized samples were re-dried at 90°C to 0 percent moisture content immediately before any analyses using oven-dry peat were conducted, to ensure test results are on a dry basis. (An alternate method is to test for moisture content on parallel samples at the time of individual dry test, with a subsequent recalculation to a dry weight basis.)

Sample not required for additional analyses was bottled, and retained as backup reference material.

2.11 ASH CONTENT

The ash content indicates accumulation of mineral matter as a result of decomposition and sediment load in peatland water (after NRCC 1979; MDNR 1982; ASTM D2974-71; Walmsley 1977). Organic soils or sediments are considered to be peat only if they contain 25 percent or less inorganic material (i.e., ash) on a dry weight basis (Andrejko *et al.* 1983).

Sample: 1 g at 0 percent moisture content.

2.11.1 PROCEDURE

Weigh a 1 g oven-dry sample to the nearest 1 mg.

Place in a porcelain crucible.

Ignite in a muffle furnace at 750°C for 30 minutes, remove from furnace for 5 minutes to ensure sufficient oxygen supply, and continue to ash for a further 30 minutes (i.e., 1 hour total ashing). Check each sample after ashing to ensure it is completely ashed, with no charred material remaining. Otherwise, stir charred portion to the top of the ash, and continue ashing.

Cool in a dessicator.

Reweigh to the nearest 1 mg.

Ash content on a percentage basis, to the nearest 0.1% oven-dry peat

$$= (F \times 100)/G$$

where F = grams of ash,
G = grams of oven-dry, homogenized samples.

Note: Organic matter (%) can be calculated by difference

$$= 100 - \% \text{ ash.}$$

2.12 VOLATILE MATTER

Volatile matter is expressed as the percentage of gaseous fraction obtained by combusting a peat sample; relates to the reactivity of peat to some processing methods. A high volatile matter content results in reduced combustion temperature (Sheppard 1984).

Sample: 1 g at 0 percent moisture content. Result: volatile matter, percent oven-dry peat. The method is ASTM D3175-77, Volatile Matter in the Analysis of Samples of Coal and Coke.

2.12.1 PROCEDURE

Weigh a 1 g oven-dry sample to nearest 0.001 g, transfer into a pre-weighed platinum crucible, and cover the crucible.

Insert into tube furnace maintained at 950°C ± 20°C, and lower sample immediately to the 950 zone. A nitrogen flush, at a flow rate of 10 ml/minute, prevents sparking of the samples.

Heat for exactly 7.0 minutes, then remove crucible from furnace and allow it to cool in a dessicator, without disturbing the cover.

Weigh as soon as cool (a few minutes cooling).

2.12.2 CALCULATIONS

Volatile Matter (%) = $(A-B)/A \times 100$

where A = weight of dry sample used (g),
B = weight of sample after heating (g).

2.13 CALORIFIC VALUE

Sample: 1 g at 0 percent moisture content. Result: net calorific value, cal/g (1 BTU/lb = 0.55 cal/g). The U.S. Department of Energy considers peats with gross calorific values of 4400 cal/g or more as fuel grade peat (LeMasters *et al.* 1983). At average levels of N (1.5%), S (0.18%), and H (5.0%), this value corresponds to a net calorific value of about 4165 cal/g for fuel grade peats.

Press a 1 g sample (oven-dry) into a 6 mm diameter pellet using a hydraulic press at 30,000 pounds per square inch.

Store the pellet at 90°C until analysis.

Reweigh the sample prior to analysis. The pressed sample's weight is the weight of sample used for the calculations.

Determine the calorific value in the pelletized sample according to ASTM D2015-77, using a Parr Oxygen Bomb Calorimeter Model 1221. An outline of the method follows.

Add 1 ml of water to the bottom of the bomb and place the pelletized peat sample and a 10 cm length of firing wire into the bomb. Seal tightly and fill with O_2 to 400 pounds per square inch.

Place bomb in water jacket and seal tightly.

Allow jacket temperature to equilibrate, then record the initial temperature (to 0.01°C).

Ignite sample, and time precisely 7.0 minutes. Record the temperature to 0.01°C.

Check that sample has been completely burned.

Gross calorific value can be calculated from the difference in temperature ($T_f - T_i$), using cal/°C values measured in standardization runs with benzoic acid, and corrected for length of firing wire burned.

Gross calorific values were corrected for %N, %S, and %H by:

Net Calorific Value
= Gross Calorific Value
- (%N x 10.3 x wt. sample)
- (%S x 13.2 x wt. sample)
- (%H x 51.03 x wt. sample).

All reported sample values are net calorific values.

"Net calorific value" is used by Finland's Technical Research Centre to refer to the gross value minus the energy used to vapourize water present in the sample and used to vapourize water formed during combustion from hydrogen present (assumed to be at a level of 5.6 percent hydrogen). This "net" value also reflects compensation for nitrogen or sulphur in terms of production of nitric and sulphuric acids in the bomb calorimeter (Sheppard 1984).

2.14 TOTAL CARBON

Total inorganic and organic carbon.

Sample: 0.05 peat at 0 percent moisture content.
Result: Total carbon, percent dry peat.

Weigh a 50 mg sample of oven-dry peat into a ceramic crucible. Add Leco iron and tin chip accelerators. Ignite in a Leco Induction Furnace under a stream of oxygen (temperature >1500°C) for 1 to 1 1/2 minutes. Carbon in the peat is converted to CO_2 and CO; sulphur, nitrogen, and hydrogen are also oxidized into gases.

The resulting gas is passed through a dust trap, then a MnO_2 trap which removes SO_x and NO_x, and then a CuO converter which catalyzes the conversion of CO to CO_2.

The CO_2 content is measured volumetrically by determining the volume of gas absorbed by a KOH solution.

2.15 ORGANIC CARBON

Sample: 0.05 g peat at 0 percent moisture content.

Result: organic carbon, percent dry peat.

2.15.1 PROCEDURE

Weigh a 50 mg oven-dry sample into a 250 ml beaker.

Add 20 ml of HCl and 80 ml of deionized H_2O.

Digest on a hot plate for 1 hour in covered beaker.

Add 1 ml of nitric acid and filter through a glass fiber filter.

Wash with deionized water.

Dry the residue and determine the remaining carbon (organic carbon) content by the method used for total carbon (Section 2.14).

2.16 NITROGEN

Sample: 0.1 g peat at 0 percent moisture content.

Result: nitrogen, percent dry peat.

Nitrogen is determined on a 0.5 to 1.0 g sample of oven-dry peat by Kjel-Foss Automatic 16200 nitrogen determinator (or by the Semi Micro-Kjeldahl Method (NRCC 1979) if the apparatus is unavailable).

2.17 HYDROGEN

Sample: 0.1 g peat at 0 percent moisture content.

Result: hydrogen, percent dry peat.

Weigh a 100 mg sample of oven-dry peat into a Leco 528-038 crucible.

Add 1 g of granular tin, 1 g of iron powder, and 0.3 g copper into the crucible and mix.

Ignite in a Leco Induction Furnace under a stream of oxygen for 2 to 5 minutes (longer if moisture still visible in the elbow of the combustion tube), at a flow rate of 0.2 to 0.25 l/minute. (The temperature setting on the Leco is at low or medium.)

The resultant water is trapped by a magnesium perchlorate absorption tube (with dust filters). Allow the gas to pass through the system for 5 minutes (only the first time for each particular magnesium perchlorate trap), disconnect the absorption tube and weigh (to give the blank value). Replace the tube again in the outlet and run for 2 minutes or until no more water is visible in the elbow of the combustion tube. Remove absorption tube and weigh.

In this case, the increase in weight x 10 x 0.1119 x 100 = $\%H_2$.

Absorption tubes can be used for up to 30 percent of the weight of magnesium perchlorate.

To ensure a source of water-free oxygen, the oxygen stream was passed through a regulated system consisting of a flow gauge, H_2SO_4 trap, glass wool dust filter, followed by a magnesium perchlorate trap. The oxygen used must be free of water. If the oxygen has a level of 750 ppm H_2O (= 99.93 pure O_2), then the overestimate of H_2 values will be 1.0 percent H_2, based on a flow of 1.2 to 1.5 l/minute for 7 minutes. Oxygen impurities may also be compensated for by blank runs.

Also, note that an average of 10 percent H_2O in a peat sample will result in an overestimate of the reported hydrogen on the basis of:

$$\%H\ (\text{dry}) = \frac{\%H\ (\text{wet}) - (2/18 \times \%H_2O)}{1 - \frac{\%H_2O}{100}}$$

Variation in H values subsequently affects calorific values by ca. %H x 51.03 x samples weight in grams (*see* Section 2.13).

2.18 SULPHUR

Sample: 0.1 g peat at 0 percent moisture content.

Result: sulphur, percent dry peat.

Weigh a 100 mg sample into a Leco 528-038 crucible.

Add 1 g of granular tin, 1 g of low sulphur iron powder, and 0.3 g of copper into the crucible and mix.

Ignite in a Leco Induction Furnace under a flow of oxygen for about 7 minutes.

Pass the gas stream (SO_2) through a dust trap.

Bubble the gas stream through a 0.01 percent solution of hydrochloric acid containing potassium iodide indicator and starch.

Titrate the solution with standardized 0.10 N potassium iodate.

2.19 OXYGEN

Result: oxygen, percent dry peat.

Oxygen is calculated by difference.

$$\%O = 100 - \% \text{ (Ash + Total C + N + H + S)}$$

2.20 C:N RATIO

This value is calculated using the values obtained from the total carbon and nitrogen determinations.

2.21 MULTIELEMENT INDUCTIVELY COUPLED ARGON PLASMA EMISSION SPECTROMETER (ICAP) ANALYSIS

2.21.1 1984 WET-ASHING METHOD OF SAMPLE PREPARATION

Results: aluminum, calcium, copper, iron, lead, magnesium, manganese, phosphorus, potassium, zinc, as ppm dry peat.

The dissolution technique is a modification of the wet-ashing method of Arafat and Glooschenko (1981).

Weigh a 250 mg sample of oven-dry peat (0 percent moisture content) into a 100 ml teflon beaker.

Add 5 ml concentrated HNO_3, 5 ml concentrated HF, and 2.5 ml concentrated H_2SO_4.

Let stand for 30 minutes.

Add 3 ml concentrated HCl. Let stand 10 minutes, then add a further 2 ml HCl.

Heat mixture slowly to 100°C for 30 minutes. Increase temperature to 200°C and continue to heat for 1 1/2 to 2 hours until 2.5 ml remains (this consists of the peat sample dissolved in H_2SO_4).

Cool for 1 hour.

Add 1 ml 30 percent H_2O_2, wait for the organic digestion to subside, then add another 1 ml H_2O_2.

Add 1 ml concentrated HCl and make up to 25 ml volume with 5% HCl.

Determine the elements Al, Ca, Cu, Fe, Pb, Mg, Mn, P, K, and Zn simultaneously using Inductivity Coupled Argon Plasma Emission Spectrometer (ICAP). (The instrument used was a Jarrell Ash Model 975 Atom Comp.)

Samples were aspirated into the ICAP using a Teflon Babbington nebulizer, assisted by a Gilson peristaltic pump.

The analytical lines were as follows:

Ca	4226.73 nm
P	2136.18 nm
K	7664.91 nm
Al	3961.52 nm
Fe	2599.40 nm
Pb	2203.51 nm
Mn	2576.10 nm
Mg	2795.53 nm
Cu	3247.54 nm
Zn	2061.91 nm × 2

Interelement interferences were determined and applied to correct the values of each particular element.

The order of sample runs and corrections is indicated in Section 5.11.

2.21.2 1985 DRY-ASHING METHOD OF SAMPLE PREPARATION

A. Results: aluminum, calcium, iron, magnesium, manganese, phosphorus, potassium, zinc, as ppm dry peat.

This dissolution technique is a modification of methods currently in use for whole rock analyses.

Press 10 g of oven-dry peat (0 percent moisture content) at 3000 pounds per square inch into a briquet of 3 cm diameter.

Weigh the briquet in a previously weighed ceramic crucible; break up the briquet slightly.

Dry-ash the briquet at 400°C for 1 hour in a muffle furnace, remove and expose to air for 3 minutes, rotate tray and replace in furnace for another 1 hour. Weigh the resultant ash after the sample has cooled (ca. 2 hours).

Mix 200 mg of ash with 1.0 g of boric acid and 0.5 g of lithium carbonate in a graphite crucible. Fuse the sample and flux at 1350°C.

Dissolve the resultant material in 50 g of 6:1 H_2O:HNO_3.

Shake for 4 hours on a reciprocating shaker.

Dilute to 120 g gravimetrically with deionized water. (The equivalent weight of the sample is calculated from the ash value.)

Determine the elements Al, Ca, Fe, Mg, Mn, P, K, and Zn simultaneously by ICAP. (The instrument used was a Jarrell Ash Model 975 Atom Comp.)

Samples were aspirated into the ICAP using a Teflon Legere nebulizer (Legere and Burgener 1985), assisted by a Gilson peristaltic pump.

The analytical lines were as indicated in Section 2.12.1. Interelement interferences were calculated and applied to correct the values of each element.

B. Results: lead, copper; as ppm dry peat.

Digest 100 mg of the above ash with 10 ml in a volumetric flask with deionized water.

Run the dilution on atomic absorption for lead and copper, using automatic background correction for lead. (The instrument used in this was a Varian AA, Model 1275.)

The wet-ashing method only provides a partial solution of several major elements (Al, Ca, Fe, P, and K). These elements form relatively insoluble sulphate compounds, so that reported values are based on the assumption that samples and standards will dissolve in similar proportions, and that the standards can be used to normalize the results. In comparison, the dry-ashing procedure used in 1985 was a complete digestion, requiring no subsequent normalization. In addition, the use of a Teflon Legere nebulizer in 1985 also augmented the sensitivity of the ICAP equipment.

2.22 MERCURY

Result: mercury, ppm dry peat.

Weigh a 1 g sample of peat dried at 50°C into a 150 ml beaker.

Add 10 ml of concentrated HNO_3 and 5 ml of concentrated H_2SO_4.

Digest in a water bath at 80° to 90°C for 1 hour.

Allow to cool and dilute with deionized H_2O to 20 ml.

Add 2 drops of 4 percent $KMnO_4$.

Mercury determined by cold vapour generation and flameless atomic absorption. (The instrument used was a Varian AA, Model 1275.)

2.23 ARSENIC

Result: arsenic, ppm dry peat.

Weigh a 1 g sample of oven-dry peat into a 150 ml beaker.

Add 5 ml concentrated HNO_3.

Heat to approximately 90°C for 30 minutes, until nearly dry.

Add 3.75 ml HNO_3, 1.25 ml HCl, and 5 ml deionized H_2O.

Heat to 90°C for 1 hour.

Dilute to 10 ml with deionized water.

Add anti-foaming agent to solution before adding it to borohydride in the reaction chamber.

Analyze the digest by hydride generation and atomic absorption using a hydrogen flame with entrained nitrogen. (In 1984, an Instrumentation Laboratories AA, Model 257, was used. In 1985, a Varian AA, Model 1275, was used.)

3.0 Tests of Methods

Selected tests of methods were undertaken where problems with published methodologies were identified, or where clarification was required to ensure the comparability of data with the results of other methods used for the same parameter. Detailed tests of all methods were not part of the Inventory Project, but some testing of this nature is recommended in any such program in order to confirm or improve the techniques involved.

In the following section, individual peat samples used in the tests were taken from the main bulk of sample collections. They are identified in terms of site-related data collected by standard inventory field methods (Riley and Michaud, in preparation):

1. Site; e.g., 31C-511 indicates the peatland's index map number (511), on the 1:250 000 scale National Topographic System Map (31C) used as a base for the peatland index map;

2. Sample Location; e.g., B3900N indicates the point where a core was taken on the baseline transect 3900 m north of transect's point of origin; L828S + 200W indicates point where a core was taken 200 m west of the baseline on the sideline transect intersecting the baseline at B828S;

3. Degree of Humification; von Post scale;

4. Peat Type: field determination of botanical composition expressed as percentage composition (1 = 10%, 2 = 20%, etc.) of sedge peat (C), wood peat (L), general moss peat (S), "brown moss" peat (Sb), and sphagnum moss peat (Ss).

3.1 CATION EXCHANGE CAPACITY

3.1.1 VOLUME OF ALCOHOL WASHINGS

In the method used in this study (NRCC 1979, p.113), the ammonia acetate in excess of that which is used to displace and saturate the exchangeable cations is subsequently removed from the peat with 60 percent industrial isopropyl alcohol. In order to assess whether the prescribed four alcohol washes are sufficient to remove all excess extractant, which might otherwise be reported as NH_4^+–Nitrogen, a number of multiple washings were analyzed for their N content to assess whether equilibrium was achieved after four washes (Table 1).

The measured level of N in the ethyl alcohol was established by the laboratory to be less than 10 ppm (<0.001%). The level of N in the KCl was less than 10 ppm (<0.001%). The continued release of N into the successive alcohol washes suggests that, after about four washes, the N released into the washes is no longer NH_4OAc, but may be N contained in particulate matter physically moved into the alcohol through the filter paper, and which conferred a visible turbidity to the alcohol washes. More than four alcohol washes are not recommended under any circumstances, and should in themselves be more rigorously quantified in order to ensure comparabilty of results.

3.1.2 BLANKS

Blanks were run (n = 3) following the CEC procedure but without using peat (i.e., add NH_4OAc to filter, wash with alcohol, leach filter with KCl, etc.). This was done to try to assess any other possible sources of N in the procedure.

TABLE 1. NITROGEN CONTENT OF ALCOHOL WASHINGS (%N).

Site	41P-8	31C-511	31C-511	31C-569	42B-31	42B-31	42B-31	42B-39
Sample	B3900N	L828S+200W	L828S+200W	F900N	B1100N	B1100N	B1100N	B2500S
von Post	3	3	5	3	1	2	4	3
Peat Type	C1L1S8	Ss0	C4Ss6	L0	Ss0	Ss0	L1C3Sb7	C2Ss8
Wash 1	.111	.050	.058	.054	.0793	.0808	.0936	.0264
2	.009	.019	.014	.022	.0307	.0264	.0128	.0120
3	.002	.018	.018	.000	.0144	.0152	.0112	.0088
4	.002	.020	.014	.016	.0088	.0096	.0104	.0088
5					.0088	.0104	.0104	.0088
6					.0104	.0104	.0088	.0080
7					.0104	.0104	.0088	.0080
8					.0104	.0104	.0088	.0080
9					.0104	.0104	.0088	.0080
%N in CEC aliquot after final wash	.014	.009	.018	.026	.0108	.0081	.0096	.0107

The %N of the measured aliquot averaged 0.002 percent (0.001 to 0.004). A Kjel-Foss Automatic 16200 nitrogen determination was used. This test should be viewed in the light of N levels in the alcohol and KCl (<0.001% each).

However, assuming a 25 g fresh peat sample at 80 percent moisture content, this level (%N = 0.002) is equivalent to a CEC of about 7 meq/100g. Therefore, this level of variability is expected in determined CEC values, independent of replicate heterogeneity or methodological variations. This may also contribute to the high relative standard deviations of replicate tests of CEC done in this project (*see* Section 4.0). These low blank levels of N were not subtracted from the reported values.

3.2 pH in H_2O

The effect of adding more than 20 ml water to the 20 g peat sample was measured for several peats. Addition of up to 120 ml of water instead of 20 ml increased pH slightly as indicated in Figure 2.

3.3. CONDUCTIVITY

The effect on conductivity of adding increasing amounts of water was determined for several peats. As can be seen from Figure 3, the volume of water added has a substantial influence on the measured conductivity. Quadrupling the volume of water decreased conductivity to less than half its original value. The extra water diluted the salts, resulting in a decrease in conductivity. For one very mossy sample, addition of extra water increased conductivity, possibly because more salts were extracted from the peat by the extra water present. Calculated from these data, a factor of 2.4 was used in order to normalize values measured with 80 ml water added to values measured with 20 ml water (*see* Section 2.4).

3.4 FIBER CONTENT

There is considerable literature on the suitability and reproducibility of various measures of fiber content as a means of characterizing the degree of decomposition of peat (*see* Levesque and Dinel 1977; McDonnell and Farrell 1984).

Levesque and Dinel (1977) compared four different methods of determining fiber content of peat materials. In discussing the traditional volumetric "syringe method", they pointed out difficulties in achieving uniformity and reproducibility with this technique. Because of the large numbers of samples requiring fiber content determinations and the general inexperience of commercial laboratories with the syringe method, it was decided to use one of the techniques proposed by Levesque and Dinel (1977) which entails mechanical dispersion of the peat prior to sieving. Both the dispersion and the subsequent washing of materials on the sieve could be better quantified using one of those methods. Although mechanical dispersion for 16 hour periods was recommended by Levesque and Dinel (1977) for best equivalence of resultant data with "rubbed fiber" determinations, the constraints on using that time period for more than 2000 samples dictated a shorter period of mechanical stirring (10 minutes).

Testing this procedure against data achieved through the use of the "syringe method" confirmed a strong correlation of percent fiber content (mechanical stirring method) with percent unrubbed fiber content (syringe method), and suggests a means of equating data achieved by the mechanical stirring with values achieved by the syringe method for both "rubbed" and "unrubbed" fiber content.

Fiber contents for 12 peat samples were measured in triplicate in our laboratory by the method described in Section 2.5. As-received, wet subsamples of these peats were sent to the Land Resource Research Institute Laboratories (LRRI) of Agriculture Canada in Ottawa, where they were measured for rubbed and unrubbed fiber content by the syringe method (NRCC 1979, p.45). Three different operators evaluated the rubbed and unrubbed fiber content of each sample.

There was a consistent relationship between the OGS values for fiber content and the LRRI laboratory's results on percent rubbed fiber (Figure 4). It can be described by the line equation,

$$y = 0.713x + 30.7$$

where y = percent fiber (mechanical stirring method),

x = percent rubbed fiber (syringe method).

For samples with high fiber content, the two methods yielded similar results, while for low fiber samples, the values determined by the OGS method were generally higher.

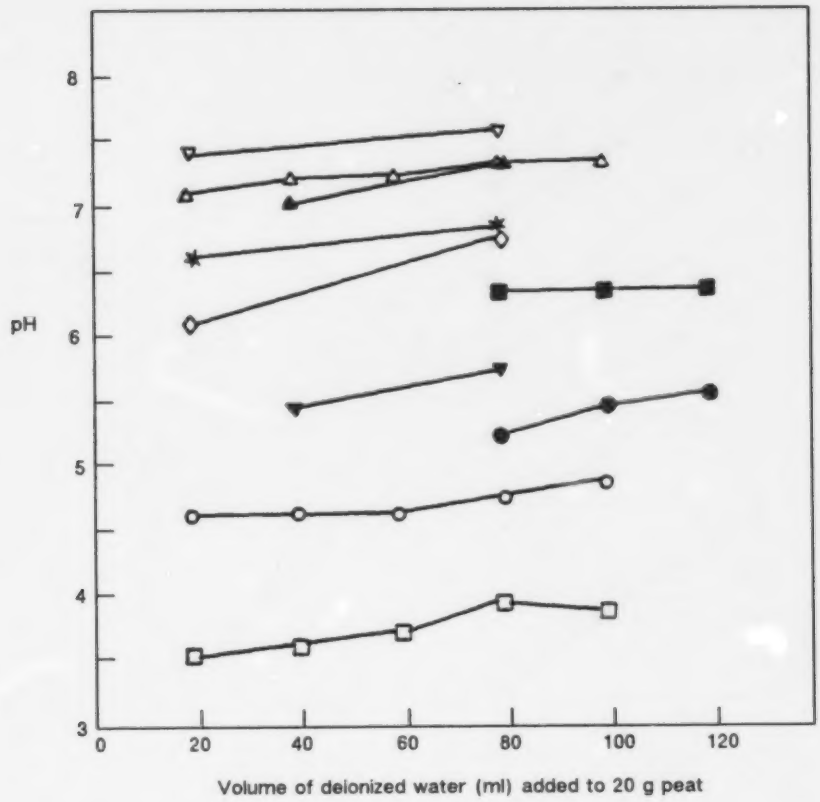
There was a much more direct correlation between the percent fiber values by the mechanical stirring method and the percent unrubbed fiber values by the syringe method (Figure 5) and, in fact, it appears that mechanical stirring for a 10 minute period does little to further break up peat fibers. This relationship can be described by the line equation,

$$y = 1.045x - 8.7$$

where y = percent fiber (mechanical stirring method),

x = percent unrubbed fiber (syringe method).

These data confirm that the mechanical stirring method used by the OGS is more realistically equivalent to unrubbed fiber by the traditional syringe method, and that mechanical stirring for a much longer period of time (16 hours, Levesque and Dinel 1977) would be required to produce data comparable to rubbed fiber as done by the syringe method.



Symbol	Sample		Peat Type	Humification (von Post)
▽	31B-4	L4800N+200W	Sb4C6	2
△	31B-4	L3000N+200W	C0	5
★	31B-18	B1400N	C2L2Ss6	2
◇	31B-8	B4300N	C0	5
○	Test Peat B (1984)		-	5
□	31B-8	B4300N	C3Sb3Ss4	1
▲	31B-4	L2100N+1000E	C2L4S4	2
■	31B-4	L2100N+1000E	C1L2Ss7	1
▼	31B-8	L3500N+300E	L2C3Ss5	2
●	31B-18	L1200W+400S	C1Ln2Ss7	1
x	42B-175	F1200W	Ss0	2

Figure 2. *Effect on measurement of peat pH of adding increasing amounts of water to peat samples. (Solid symbols indicate fibrous samples which could not be measured when only 20 ml of water was added.)*

The standard deviations (SD) of these tests (n = 3, 12 samples) were usually lower for the mechanical stirring method (SD = 1.5 to 5.4; average 2.6), than by the rubbed fiber content by the syringe method (SD = 0.6 to 17.0; average 6.6) or by the unrubbed fiber content (syringe method) (SD = 1.7 to 12.3; average 6.1). However, these are not strictly comparable because the LRRI Laboratory had three different operators performing the triplicate tests, while the other data were produced by a single operator.

Nevertheless, these data suggest, in the same manner as indicated by Levesque and Dinel (1977), that the mechanical dispersion technique may be at least as or more reproducible than the syringe method. This is an important consideration in recommending analytical procedures.

On the basis of these preliminary tests, it is recommended that the equation above ($y = 1.045x - 8.7$) be used in relating OGS's percent fi-

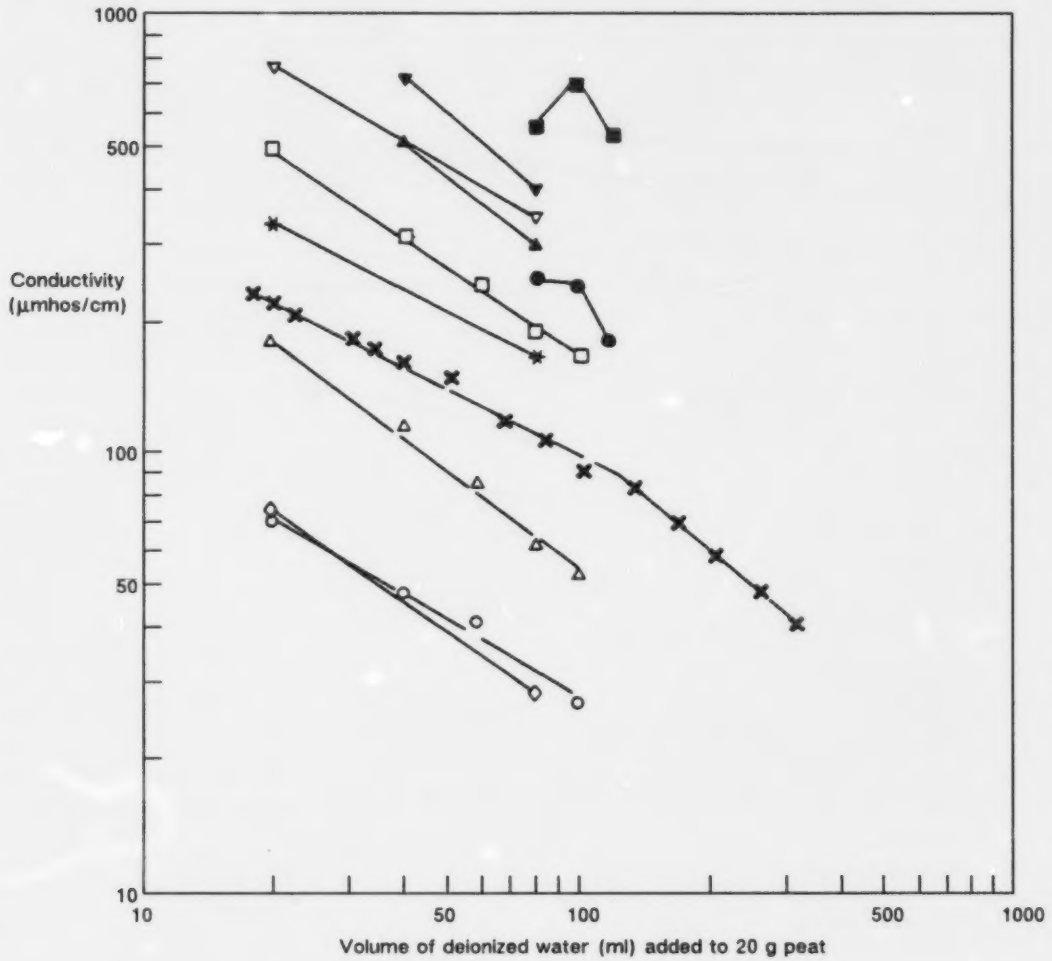


Figure 3. *Effect on measurement of conductivity of adding increasing amounts of water to peat samples. (See Figure 2 for symbol and sample explanation.)*

ber content data to other reports in the literature of percent unrubbed fiber by the syringe method. The drawback of unrubbed fiber determinations is that they normally produce values only in the 25 to 90 percent range, and do not extend to the 0 to 25 percent range achieved by the rubbed fiber determination by the syringe method (Levesque and Dinel 1977).

Therefore, in order to relate OGS's percent fiber content data to published data on percent rubbed fiber by the syringe method, a secondary equation may be used which is based on published data on both rubbed and unrubbed fiber content by the syringe method (Figure 6). The equation suggested by these data is,

$$y = 0.737x + 31.9$$

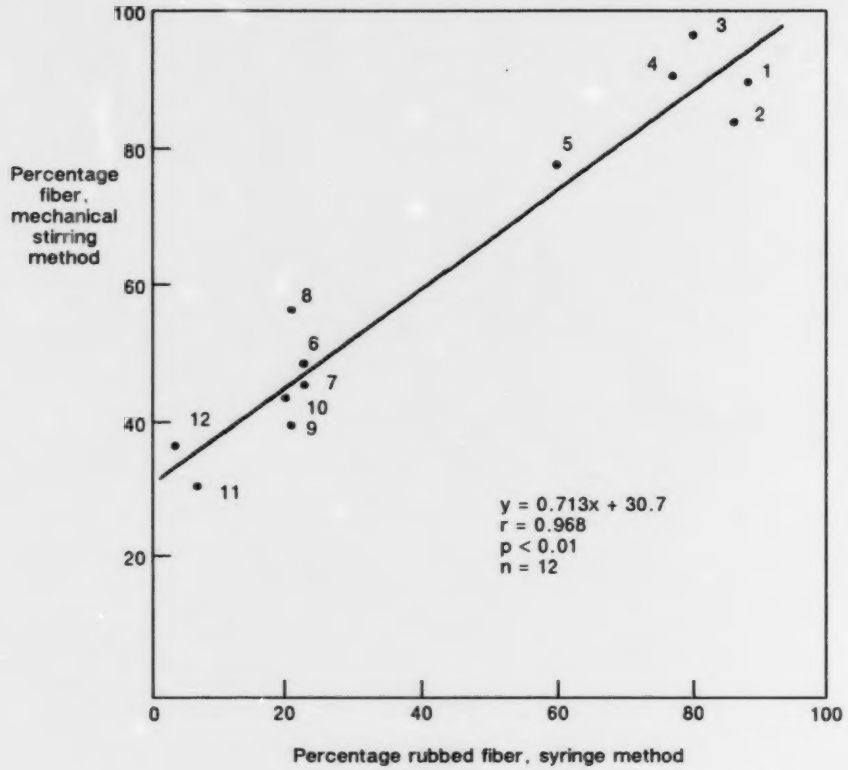
where y = percent unrubbed fiber (syringe method),

x = percent rubbed fiber (syringe method).

(Note the similarity of this line equation with that relating percent fiber content (mechanical stirring method) to percent rubbed fiber (syringe method)).

3.5 REHYDRATION OF HOMOGENIZED DRY PEAT

Ten peat samples were selected to check the rehydration rate of dry peat. Approximately 40 cm^3 of air-equilibrated peat samples, which had previously been dried at 50° or 90°C and ground through



No.	Sample		Peat Type	Humification (von Post)
1.	52G-181	B1800W	C1S9	1-2
2.	52G-191A	B1000E	C1S9	1-2
3.	42B-176	L1000S+200W	Ss0	2
4.	52G-155	B700N	Ss0	2
5.	42B-175	F1200W	Ss0	2
6.	52G-165	L000N+700W	L1Ss2C7	4
7.	52G-119	G800E+500W	C1S9	4
8.	52G-178	B1300E	C2S8	3
9.	52G-191A	L500E+3000S	S2C8	4
10.	52G-181	L400W+200S	S3C7	4
11.	52G-226	L400W+1000N	S3C6	3
12.	Test Peat B (1984)			

Figure 4. *Comparison of percent rubbed fiber (syringe method) and percent fiber content (mechanical stirring method).*

an 80 mesh sieve (*see* Section 2.10), were transferred into small aluminum dishes. Two of the samples were pressed into pellets. The samples were dried at 90°C for 2 days, then removed and covered with a second aluminum pan (not sealed) and left under room conditions. The samples were weighed immediately after drying and periodically thereafter in order to measure the rate of water absorption from the air. Room temperature ranged between 18° to 22°C, and relative humidity was 36 to 52 percent.

The results are presented in Table 2 and Figure 7 (data for pelletized samples is not plotted).

The peat samples regained moisture at a fairly slow rate, with an average rehydration of only 1.4 percent after 6 hours. They continued to gain weight over the next few days, until their moisture content appeared to stabilize at 3.5 to 9 percent after 5 or 6 days. This was less than the moisture contained in the samples after several weeks or months of equili-

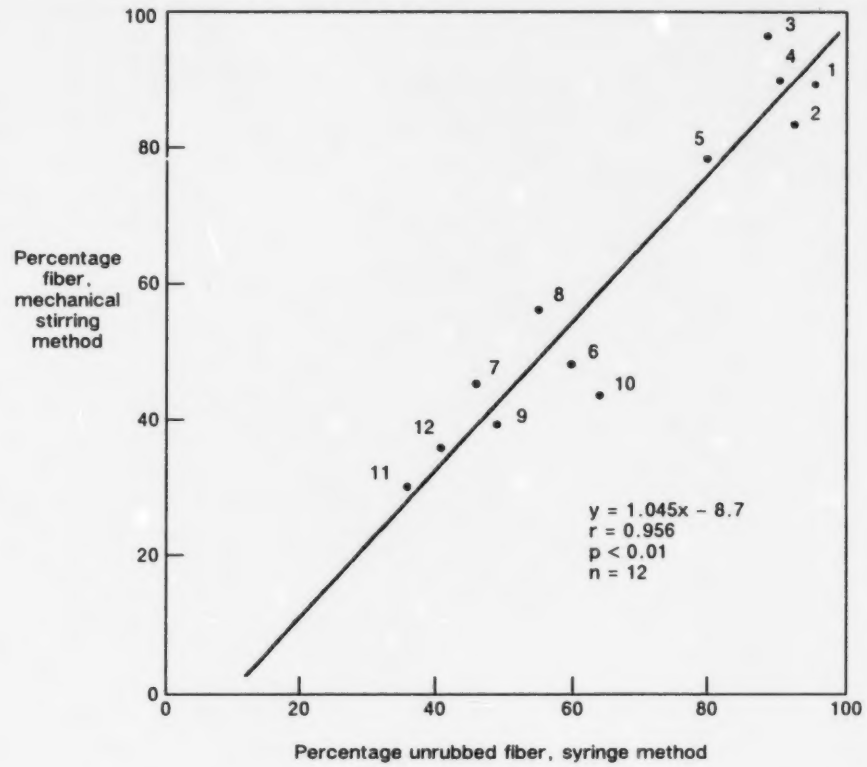


Figure 5. *Comparison of percent unrubbed fiber content (syringe method) and percent fiber content (mechanical stirring method). (See Figure 4 for sample explanation.)*

bration (9.5 to 18 percent). Data from the National Research Council Laboratory in Halifax on Ontario peat samples dried and homogenized in April 1983 and analyzed for water content in January 1984 showed a similar range of 9.9 to 13.7 percent moisture in 26 samples.

In order to determine whether the difference between percent moisture before drying and after a week of rehydration (*see* Table 4a) was due to a lack of mixing, 4 of the 8 unpelletized samples were stirred periodically from day 12 to 21. However, no increase in moisture content was seen in the stirred samples.

The small decrease in sample weight (and moisture content) seen in all samples at days 14 to 17 was correlated with a decline in relative humidity of the air ($r = 0.77$ to 0.93, $p < 0.05$ for the 8 unpelletized samples); the peat was gaining and losing moisture to the air as relative humidity fluctuated.

These results show that there is no significant rehydration if peat samples are left at room conditions for very short periods (at least in winter time). However, rehydration can alter weight significantly over several days, and samples should be re-dried if they have been left out for more than 6 to 8 hours, and for considerably less time if higher precision requirements must be met. In Section 2.10 (Sample Homogenization), it is recommended that "dried" peats be re-dried at 90°C to 0 percent moisture content immediately before test procedures using oven-dry peat.

3.6 ASH CONTENT

The ash content of several peats at different temperatures over different time periods was assessed to determine the most useful ashing regime for this study.

Six peats ranging from 5 to 65 percent ash content were ashed at temperatures of 500° to 900°C. Results are presented in Figure 8. For most peats, there was little difference in percent ash with temperature. However, one sample (31C-569, a well-decomposed woody peat) decreased in ash content from 17.3 to 17.4 percent at 500° to 550°C, to 9.1 to 12.5 percent at 650° to 900°C.

The duration of ashing had little effect after 1 hour, as Figure 9 illustrates. Only 1 sample, at

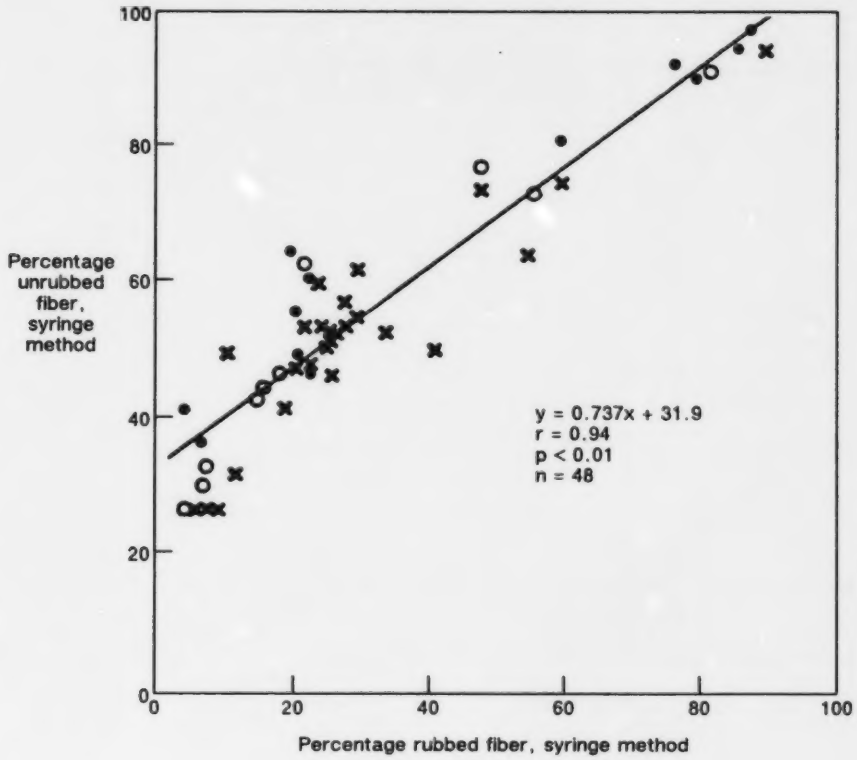


Figure 6. *Comparison of percent unrubbed fiber (syringe method) and percent rubbed fiber (syringe method).*

●	Present LRRI data	(n = 12)
○	Stanek and Silc 1977	(n = 10)
x	Levesque et al. 1980	(n = 26)

550°C, decreased in ash content by more than 10 percent between 1 and 4 hours.

Based on the above results, and on the recommendation by Andrejko *et al.* (1983) that 750°C is an appropriate ashing temperature for evaluation of peat as fuel, ashing at 750°C for 1 hour was adopted in this inventory.

Note that the removal of sample at 30 minutes to ensure sufficient oxygen supply was considered critical in achieving good results in only 1 hour.

3.7 ICAP SAMPLE PREPARATION

3.7.1 1984 WET-ASHING METHOD

Two acid digestion methods were tested to assess their suitability for analysis of minor elements by the ICAP method. As well as the method described above (Section 2.21), a nitric acid leaching was attempted. Briefly, the latter technique is as follows: 250 mg sample, add 10 ml HNO_3 (concentrated), let stand 12 hours, heat to dryness, add 1 ml HCl (concentrated), dilute to 25 ml with 5 percent HCl. Table 3a summarizes results and shows the sulphuric acid method to achieve better results.

3.7.2 1985 DRY-ASHING METHOD

ICAP results in 1984 based on wet-ashing were generally considered to lack the desired level of precision (*see* Section 4.1, 1984 Replicate and Duplicate Tests). As a result, testing was undertaken on a dry-ashing technique using an increased sample size to prepare peat samples for ICAP analysis. Results are shown in Table 3b. Additional dry-ashing tests were done on a number of peats which had been analyzed in 1984 by the wet-ashing technique. These additional blind tests showed a tendency for the dry-ashing technique to release more analyzable Ca, Al, K, Fe, and Mn (resulting in slightly higher values), and a tendency for slightly lower values for P, Mg, and Zn.

TABLE 2. REHYDRATION OF OVEN-DRY PEAT AT ROOM CONDITIONS.

Sample		Year Collected	Peat Type	von Post Humification	% Moisture before drying	% Moisture at 1 hour after drying	% Moisture at 6 hours after drying	% Moisture at 26 hours	% Moisture at 7 days
○ 52I-129	B000	1982	C3S3L3	2	11.3	0.7	1.4	3.4	8.1
□ 31F-552	B500W	1982	C0	4	16.1	0.9	1.4	3.1	9.1
△ 42G- 29	B1664W	1982	S3C7	4	9.9	1.0	1.6	3.2	7.7
▽ 31D-329	B500W	1982	L2C8	7	9.9	0.6	1.1	2.4	6.3
● 31B- 28	B3700N+1200E	1983	L2S2C6	6	13.8	0.7	1.1	2.9	7.4
■ 31C-567	G850W+400N	1983	L0	8	9.5	0.4	0.8	2.0	5.0
▲ 52D- 16	B700N	1983	Top layer	1	18.0	0.7	1.2	3.0	7.6
▼ 52D- 29	B2300W+100N	1983	Ss0	2	13.5	1.2	2.4	4.6	7.7
*31C-567	G850W+400N	1983	L0	7	-	0.2	0.4	0.9	3.5
*52D- 43	L700W+400N	1983	S3C7	2	-	0.4	0.7	1.4	5.9
Mean value for unpelletized samples					12.7%	0.8%	1.4%	3.1%	7.4%

* *These two samples were pelletized in preparation for determination of calorific value.*

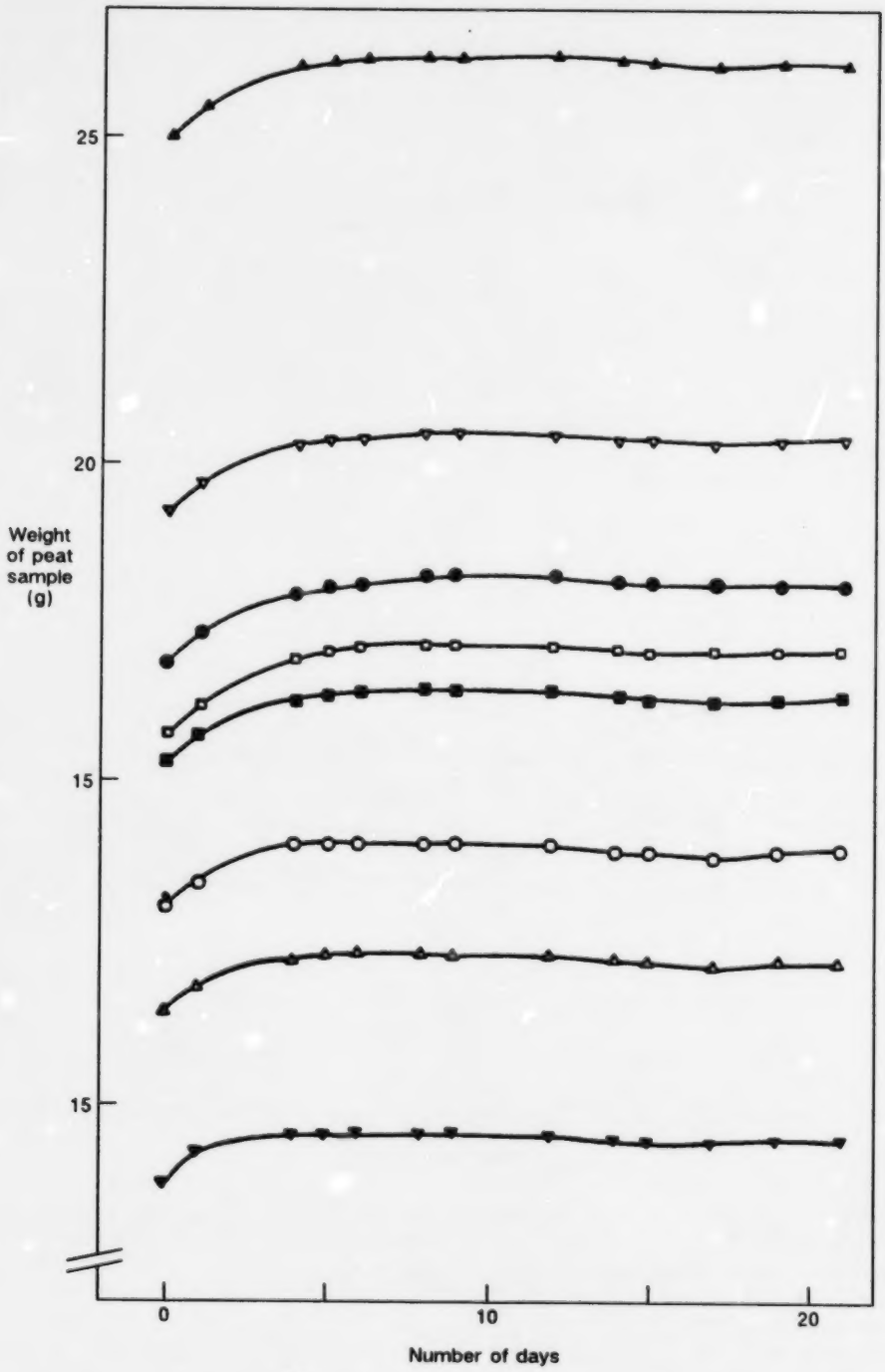
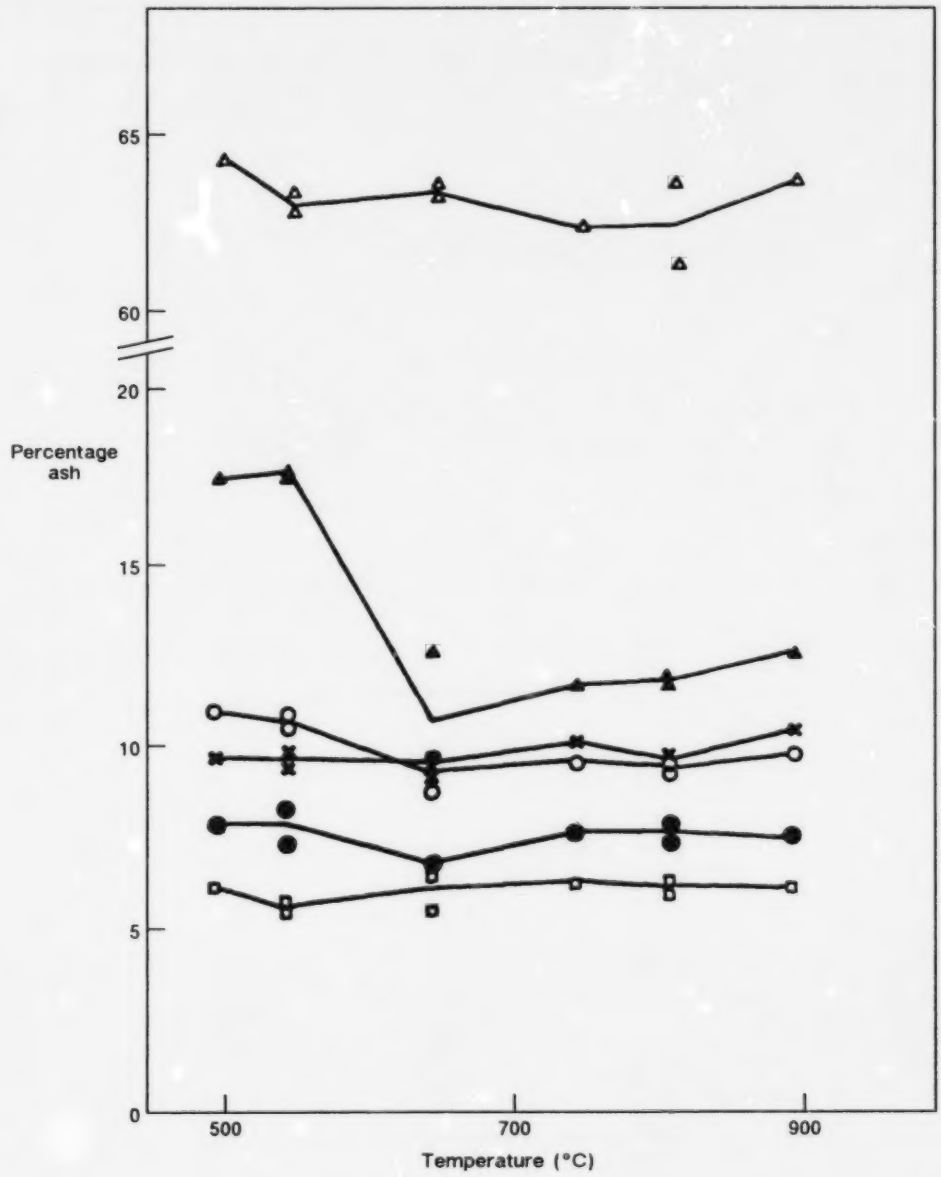


Figure 7. *Rehydration of peat samples over time. (See Table 2 for symbol and sample explanation.)*



Symbol	Sample		Peat Type	Humification (von Post)
△	31C-567	G850W+400N	L10	8
▲	31C-569	H1100N	L10	7
○	31B-8	L3500N+300E	C2L8	3
×	Test Peat B (1984)		-	5
●	31B-8	L3500N+300E	L2C3Ss5	2
□	52D-29	B2200W	Ss0	2

Figure 8. *Percent ash content after ashing at various temperatures for 1 hour.*

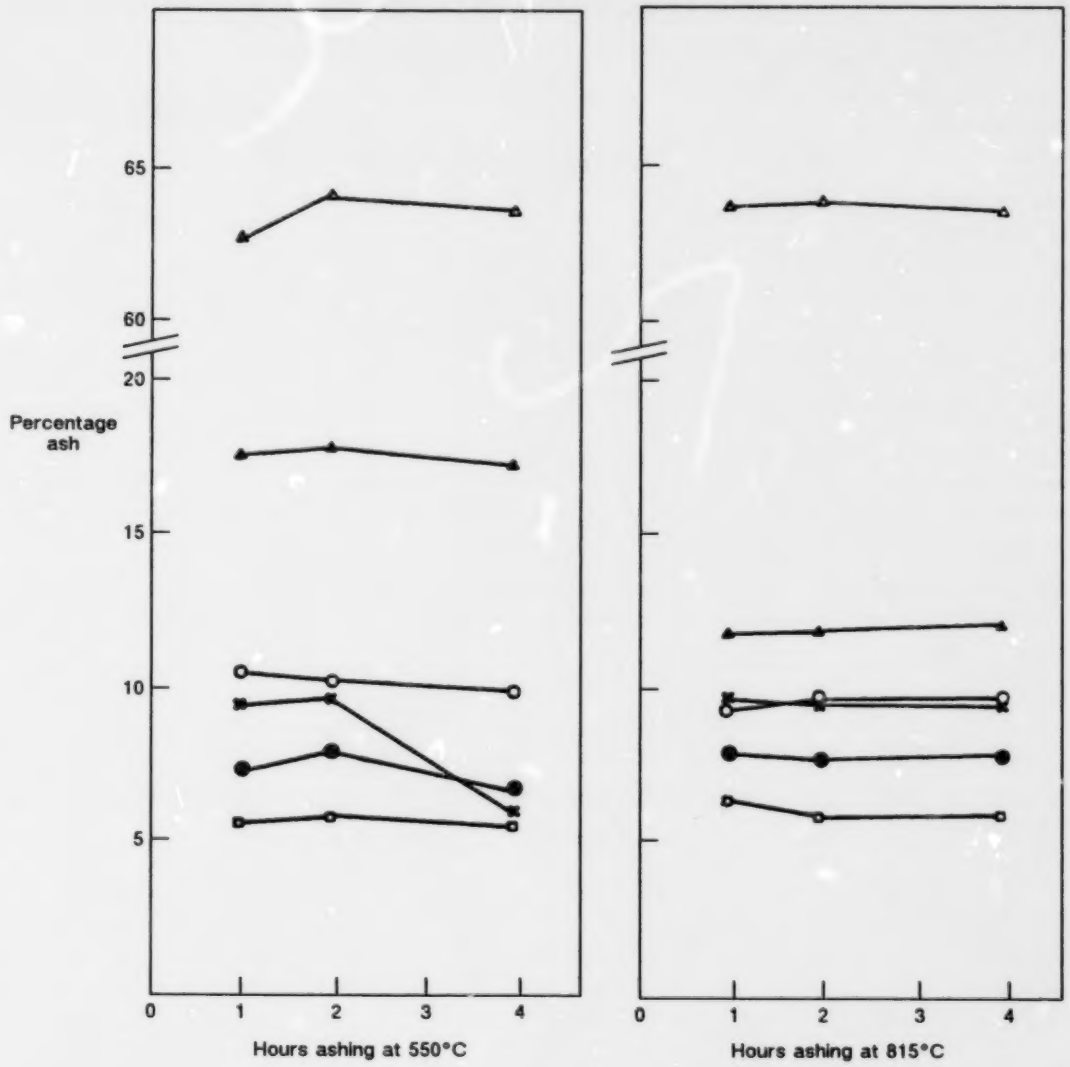


Figure 9. *Percent ash content after ashing peat for various time periods at 550°C and 815°C. (See Figure 8 for symbol and sample explanation.)*

Other laboratories undertaking similar multielement ICAP analyses are advised to undertake similar sample preparation tests to ensure that accuracy and precision meet the levels considered necessary to their particular studies.

Laboratory staff tested dry-ash samples for Cu, Pb, and Zn, both on ICAP and on atomic absorption (AA) following an aqua regia digest of the ash. The test results (Table 3c) were inconclusive, but it was decided to use ICAP for Zn, and aqua regia/AA for Cu and Pb.

During the testing of this technique, Peat A (1985) and Peat B (1985) were run to assess the precision of the results. These test results are shown on Table 3d, as are the mean values of Peat A (1985) and Peat B (1985) derived from blind replicate tests (Section 4.2). These data are useful as a check on variation between sample results done while processing test materials versus sample run materials. These data suggest that, with the exception of Pb, Cu and, to a lesser extent Zn, results were comparable between test runs and the main sample run.

TABLE 3A. 1984 TEST RESULTS OF TWO WET-ASHING TECHNIQUES FOR MULTIELEMENT ICAP ANALYSIS (ppm).

	Fe	Ca	Mg	K	Mn	P	Cu	Pb	Zn
Known Standards									
a) Sediment A	30,476	4,819	8,683	18,592	513	636	25	34	191
b) NBS 1571	300	20,900	6,200	14,700	91	2,100	12	45	25
c) NBS 1575	200	4,100	---	3,700	675	1,200	3	11	--
Nitric Digestion									
Mean - Sed. A (n = 2)	20,302	2,239	4,923	4,126	216	519	18	42	148
Mean - 1571 (n = 5)	239	18,329	4,474	14,517	65	1,805	8	53	15
SD - 1571	20	609	104	465	2	65	0.5	7	4
Mean - 1575 (n = 5)	175	3,804	975	3,610	499	1,068	1.2	9	61
SD - 1575	10	36	9	61	2	17	0.4	1	6
% of Sed. A Standard	67	46	57	22	42	82	72	122	77
% of NBS 1571	80	88	72	99	71	86	70	117	62
% of NBS 1575	88	93	--	98	74	89	40	85	--
Sulphuric Digestion									
Mean - Sed. A	28,508	3,057	6,517	15,451	395	700	25	36	176
Mean - 1571	368	14,292	4,923	12,617	69	2,047	11	41	24
SD - 1571	67	2,631	145	444	2	62	0.5	7	1
Mean - 1575	224	4,064	1,080	3,298	541	1,255	3	15	71
SD - 1575	13	267	40	101	20	27	0	4	13
% of Sed. A Standard	94	63	75	83	77	110	100	106	92
% of NBS 1571	123	68	79	86	76	97	95	90	97
% of NBS 1575	112	99	--	89	80	105	100	135	--

TABLE 3B. 1985 TEST RESULTS OF DRY-ASHING TECHNIQUE FOR MULTIELEMENT ICAP ANALYSIS (ppm).

	Al	Fe	Ca	Mg	K	Mn	P	Zn
Known Standards								
a) OGS 1878P	7,500	10,000	43,000	4,100	2,800	240	1,800	48
b) NBS 1575	545	200	4,100	-	3,700	675	1,200	-
OGS 1878P								
Test Mean (n = 5)	7,926	10,680	46,860	4,234	3,164	270	1,822	33
SD	62	110	404	31	67	2	13	1
RSD	1%	1%	1%	1%	2%	1%	1%	3%
% of Standard Value	106%	107%	109%	103%	113%	113%	101%	65%
(1984 values (n = 4) by wet-ashing)	(5,539)	(10,026)	(39,056)	(4,672)	(-)	(240)	(2,067)	(46)
NBS 1575								
Test Mean (n = 5)	604	207	4,402	1,094	3,718	635	999	55
SD	14	8	88	22	79	13	20	5
RSD	2%	4%	2%	2%	2%	2%	2%	8%
% of Standard Value	111%	104%	107%	-	101%	94%	83%	-

TABLE 3C. 1985 TEST RESULTS OF DRY-ASHING TECHNIQUE FOR Cu AND Pb BY AQUA REGIA/ATOMIC ABSORPTION (ppm).

	Cu	Pb	Zn			
Known Standard Values						
a) OGS 1878P	196	82	48			
b) NBS 1575	3	10.8	-			
		ICAP			Aqua Regia/AA	
	Cu	Pb	Zn	Cu	Pb	Zn
OGS 1878P						
Test Mean (n = 5)	198	68	33	207	56	51
SD	8	10	1	10	4	4
RSD	4%	15%	3%	5%	7%	8%
% of Standard Value	101%	83%	69%	106%	68%	106%
NBS 1575						
Test Mean (n = 5)	17	30.8	55	22	9.6	86
SD	5	1.1	5	19	.9	22
RSD	29%	4%	8%	86%	9%	26%
% of Standard Value	573%	285%	-	730%	87%	-

TABLE 3D. 1985 TEST RESULTS OF DRY-ASHING/ICAP ANALYSIS (ppm) OF BLIND TEST PEATS A AND B (1985) IN COMPARISON WITH RESULTS ON SOME PEATS RUN AS BLIND REPLICATES WITH THE MAIN SAMPLE RUN (ppm).

				ICAP					AA	
	Ca	P	K	Al	Fe	Mn	Mg	Zn	Pb	Cu
Blind Replicate Test Results										
Peat A (1985) (n = 25)	2,460	1,857	1,556	11,220	2,200	39.6	507	66.7	34.6	10.1
RSD	9%	9%	10%	9%	11%	14%	4%	10%	39%	27%
Peat B (1985) (n = 25)	2,058	887	1,029	15,063	1,808	20	521	5.6	4.4	4.7
RSD	5%	4%	7%	3%	3%	6%	3%	45%	84%	45%
Peat A (1985)										
Test Value (n = 8)	2,404	1,767	1,733	10,498	2,071	39.1	525	68.5	57.9	25.5
SD	111	132	172	286	199	2.2	28	4.5	5.0	1.7
RSD	5%	8%	10%	3%	10%	6%	5%	7%	9%	7%
% of Replicate Value	98%	95%	111%	94%	94%	99%	104%	103%	167%	253%
Peat B (1985)										
Test Value (n = 8)	2,323	969	1,131	13,757	1,823	19.3	478	11.9	7.7	10.9
SD	62	67	85	303	48	1.9	14	1.9	1.9	0.4
RSD	3%	7%	8%	2%	3%	10%	3%	17%	25%	3%
% of Replicate Value	113%	109%	110%	91%	101%	97%	92%	213%	175%	232%

4.0 Duplicate and Replicate Test Results

4.1 PRECISION REQUIREMENTS

Two sets of blind replicate peat samples were incorporated into the peat samples sent for analysis. In each year of analysis, an unhumified peat (Peat A, H2-3) and a humified peat (Peat B, B4+) were split into numerous samples (34 and 46 in 1984, 25 and 25 in 1985), and the required physical and chemical tests were conducted during the course of the general laboratory analysis of the bulk of the samples. The Peats A and B in 1985 were different than those used as Peats A and B in 1984. (Difficulties inherent in trying to ensure or assess the homogeneity of replicate peat samples occur when they are prepared from large bulk samples of wet peat.)

A "required practical determination limit" (PDL, *see* Tables 4b and 4d) was established for each parameter tested. These were set on the basis that, in order to obtain data suitable for sound statistical manipulation, the lowest anticipated level of occurrence in the sample should equal or exceed 10 x PDL. For example, mercury data were anticipated to be of significance at and above 0.1 ppm; accordingly, a required PDL was set at 0.01 ppm. In some cases, this criterion was compromised by the lack of sensitivity in the method available.

The precision requirements and assessments were made in terms of the 95 percent confidence limit criterion. For all values above 10 x PDL, the 95 percent confidence limits were set at 10 percent of the mean reported value (i.e., for replacement data sets, $2\sigma \leq 0.10\ x$, where σ is the standard deviation and $\bar{x}$ is the mean). For replicate data sets, the allowable difference (Δ) from the mean ($\bar{x}$), when the mean is less than or equal to 10 x PDL, is 1 x PDL. If the mean is greater than 10 x PDL, the difference from the mean must not exceed $0.1 \times \bar{x}$.

A factor analysis was performed on each member of the replicate set with respect to the data set's mean. To exclude the inevitable anomalous results, any member of the data set more than 2 x Standard Deviation (SD) from the mean of the data set was omitted, and a subsequent mean and SD were calculated.

EXAMPLE 1.

A. Assume PDL = 1 ppm, and
$\bar{x}$ = 12.5 ppm,
based on the following data set:

10	(Δ = 2.5
15	2.5
12	0.5
13	0.5)

Because $\bar{x}$ is greater than 10 x PDL (12.5 > 10.0),
$0.1 \times \bar{x}$ = 1.25
= 1.3.

Factor Analysis Criteria:

1. Values acceptable if $\frac{\Delta}{0.1 \times \bar{x}} \leq 1$;
2. Values marginally acceptable if $\frac{\Delta}{0.1 \times \bar{x}} \leq 1.5$;
3. Values unacceptable if $\frac{\Delta}{0.1 \times \bar{x}} > 1.5$;

Therefore:

$\frac{2.4}{1.3} = 1.9$; $1.9 > 1.5$; values unacceptable;

$\frac{0.5}{1.3} = 0.4$; $0.4 < 1.0$; values acceptable.

50 percent of the above data set is acceptable.

B. Assume PDL = 2 ppm, and
10 x PDL = 20 ppm,
based on the same data set as above.

Because $\bar{x} < 20$, then Δ must not exceed 2.

Therefore:
2.5 > 2, and data set members 10 and 15 are unacceptable;
0.5 < 2, and members 12 and 13 are acceptable.
Again, 50 percent of the data set is acceptable.

Blind duplicate sample pairs (46 in 1984, 21 in 1985) were also included throughout the peat samples analyzed. The allowable difference between duplicates is 15 percent, at 95 percent confidence limits. If the mean exceeds 10 x PDL, the difference (Δ) between the duplicate values must not exceed $0.15 \times \bar{x}$, where $\bar{x}$ is the mean of the individual duplicate pair. Again a factor analysis was applied.

EXAMPLE 2.

A. Assume PDL = 10, and
$\bar{x}$ = 109,
based on the duplicate pair: 100
118.
Because $\bar{x}$ > 10 x PDL (109 > 100),
$0.15 \times \bar{x}$ = 16.4.

Factor Analysis Criteria:

1. Values acceptable if $\frac{\Delta}{0.15 \times \bar{x}} \leq 1.0$;
2. Values unacceptable if $\frac{\Delta}{0.15 \times \bar{x}} > 1.5$;
3. Values marginally acceptable if $\frac{\Delta}{0.15 \times \bar{x}} \leq 1.5$;

In this case, $\frac{\Delta}{0.15 \times \bar{x}} = \frac{18}{16.4} = 1.1$, and

1.0 < 1.1 < 1.5; then the duplicate set is marginally acceptable.

(For a duplicate set whose mean value is less than 10 x PDL, the difference between duplicate

values must not exceed 1.5 x PDL to be acceptable.)

B. Using the above duplicate data, and a PDL = 20, $\bar{x}$ = 109, $\bar{x}$ < 10 x PDL (109 < 200).

In this case, 1.5 x PDL = 1.5 x 20 = 30.
Then, because 18 < 30, this pair is acceptable.

A total of acceptable, marginally acceptable, and unacceptable pairs for each test was obtained and expressed as a percentage of the total number of duplicate pairs tested.

4.2 1984 TEST RESULTS

4.2.1 RESULTS OF BLIND REPLICATE TESTS

Two peat controls were developed in 1984 for this project, Peat A (1984) and Peat B (1984). The National Research Council Laboratory at Halifax tested these peats for some parameters, but the lack of recognized peat standards means that most of the data can only be useful in assessing precision rather than accuracy.

Peat A (1984) was disguised as 45 blind replicates; Peat B (1984) as 46 blind replicates. Results are presented in Table 4a.

In light of the low relative standard deviations of the results on some important parameters, such as moisture content, volatile matter, calorific values, carbon, and pH, it is difficult to attribute some of the poorer replicate results to sample heterogeneity alone. As mentioned previously, some of the poor precision was attributed to the wet-ashing technique employed in 1984.

4.2.2 RESULTS OF BLIND DUPLICATES

Forty-six blind duplicate pairs were assessed. Table 4b summarizes the required practical determination limit (PDL) for each test, the percentage of pairs of duplicates considered acceptable by the criteria indicated in Section 4.1, and the effective PDL suggested by these data if a minimum of 80 percent acceptability is required for blind duplicate pairs.

It should be noted that the "effective" practical determination limits suggested by these data were not considered to be the lowest levels achievable by the methods outlined. This problem is also discussed in connection with ICAP results, standards, and tests of methods. Further testing of the sample preparation techniques previous to ICAP analysis, in particular increased sample size and possible dry ashing, were undertaken as a result of these 1984 data. The 1985 data reflect these changes.

4.3 1985 TEST RESULTS

4.3.1 RESULTS OF BLIND REPLICATE TESTS

Two peat controls were developed in 1985 for this project. Peat A (1985) was disguised as 25 blind replicates; Peat B (1985) as 25 blind replicates. Results are presented in Table 4c.

In terms of acceptability of the results of these blind tests, all results were improved in 1985 to a level of more than 80 percent of results acceptable or marginally acceptable, with the exception of results of lead, copper, and zinc, in which case the elements were at very low concentrations.

4.3.2 RESULTS OF BLIND DUPLICATES

A total of 21 blind duplicate pairs were assessed. Table 4d summarizes the required practical determination limit (PDL) for each test, and the percentage of pairs of duplicates considered acceptable by the criteria indicated in Section 4.1. It also indicates the effective PDL suggested by these data if a minimum of 80 percent acceptability is required for the blind replicate pairs.

These tests showed marked improvement over 1984 (Table 4b), much of it attributable to more reliable ICAP elemental analysis based on dry-ashing sample preparation. A number of tests continued to produce results at precision levels below that which was considered ideal:

1. Cation Exchange Capacity
2. Conductivity
3. Potassium
4. Copper
5. Zinc

In the case of cation exchange capacity and conductivity, the results are most likely inherent in the methods. In the case of arsenic, lead, copper, zinc, and mercury, mean test values were often at such low levels ($\bar{x}$ < 10 x PDL) that the assessment of precision is problematic anyway.

On the basis of these tests, it is suggested that the values recorded in the column entitled "PDL required to render duplicate pairs ±80% acceptable", are values which were generally achieved by the methods used in this laboratory analysis, and which should be expected to apply as practical determination levels for these methods during the course of large-scale peat analyses. These PDLs can be lowered in "small batch" tests and, as demonstrated in 1984 and 1985, can be lowered through experience with the methods and improvements in test procedures.

4.4 INTERNAL LABORATORY CONTROLS

The laboratory was directed to select two samples per test, to be subsampled as 10 individual replicates

TABLE 4A. BLIND REPLICATE TEST RESULTS—1984.

	Peat A (1984)							
Test	Mean	Range	n =	SD	% RSD	NRC Data	% Acceptable ($\Delta/A \leq 1$)	% Acceptable & Marginally Acceptable ($\Delta/A \leq 1.5$)
Cation Exchange Capacity (meq/100g)	99	53–191	33	29	29	--	33.3	48.5
Conductivity (µmhos/cm)	211	73–287	34	54	26	--	17.7	41.2
Fiber Content (%)	54.1	39–75	33	7.1	13	--	68.8	78.2
Moisture Content (%)	86.1	84.1–93	33	0.92	1	87.5	100	100
Bulk Density (dry) (g/cm^3)	0.15	0.07–0.21	34	0.02	16	--	82.4	94.2
Absorptive Capacity	12.4	10.6–14.8	33	1.1	9	--	78.8	90.9
Ash Content (%)	12.3	10.7–15.7	28	1.2	9.6	13.1	75.0	92.9
Volatile Matter (%)	65.3	65.2–68.0	29	1.3	2	62.1	100	100
Calorific Value(net) (cal/g)	4272	4112–4851	20	191	5	4906*	100	100
Carbon – Total (%)	47.1	42.0–50.4	32	1.7	4	47.5	96.9	100
Carbon – Organic (%)	43.9	39.3–49.5	32	2.8	6	--	90.6	100
Nitrogen (%)	2.3	1.8–4.0	30	0.41	18	2.1	80.0	90.0
Hydrogen (%)	4.7	4.2–5.2	20	0.38	8	4.3	95	95
Sulphur (%)	0.6	0.4–0.79	33	0.08	14	0.82	84.9	84.9
Arsenic (ppm)	<0.81	<0.1–1.9	31	<0.44	<54	--	---	---
Mercury (ppm)	0.13	0.04–0.28	34	0.06	51	--	14.7	32.4
Calcium (ppm)	3165	2480–5671	31	852	27	--	25.8	45.2
Phosphorus (ppm)	1367	1245–2065	31	172	11	--	62.7	90.3
Potassium (ppm)	1738	1351–2881	31	341	20	--	40.0	66.7
Aluminum (ppm)	6604	4211–9224	30	1350	20	--	43.3	53.3
Iron (ppm)	2454	1537–4047	31	588	24	--	38.7	48.4
Lead (ppm)	68.1	51–109	30	2.3	18	--	53.3	70.0
Manganese (ppm)	38	31–50	31	64	17	--	41.9	58.0
Magnesium (ppm)	747	484–1323	31	181	24	--	35.5	54.9
Copper (ppm)	20	13–35	31	4.3	22	--	53.3	66.6
Zinc (ppm)	91.5	63–155	31	21.0	23	--	38.7	54.8
pH–H_2O	4.0	3.5–4.3	33	0.19	5	--		
pH–CaCl	3.5	3.2–4.0	34	0.21	6	--		

** NRC values are gross calorific values, uncorrected for N, H, S. All other reported calorific values are net calorific values.*

each, to provide internal quality control for the methods. The reported data have more relevance to tests of precision than accuracy.

Those replicate sets with asterisks (*) have a relative standard deviation (% RSD) less than 50 percent of that obtained by blind replicate tests for the same year's data. Figures are all rounded to one decimal place. Samples are annotated (°) if the reported mean values are close to required practical determination limits (PDL), or if determination limits do not apply (e.g., pH) (*see* Section 4.4.1).

For the 1983 to 1984 results which are not close to the required PDLs (49/56), 65 percent (32/49) of the internal replicate sets RSDs are at levels less than 50 percent of the RSDs reported for the blind replicate tests for the same parameters. In 1984 and 1985, the comparable figure is 69 percent (31/45).

TABLE 4A. CONTINUED.

Peat B (1984)

Test	Mean	Range	n =	SD	% RSD	NRC Data	% Acceptable ($\Delta/A \leq 1$)	% Acceptable & Marginally Acceptable ($\Delta/A \leq 1.5$)
Cation Exchange Capacity (meq/100g)	104	47- 181	46	23	22	--	43.5	63.1
Conductivity (μmhos/cm)	62	40- 75	46	10.4	17	--	52.2	69.6
Fiber Content (%)	37	12- 56	44	9.1	25	41 unrubbed	44.0	55.8
Moisture Content (%)	84	79.1-91.0	46	2.7	3	87.9	100	100
Bulk Density (dry) (g/cm^3)	0.16	0.09-0.21	46	0.03	17	--	68.1	80.9
Absorptive Capacity	7.5	5.2-13.3	46	1.9	26	--	19.6	47.8
Ash Content (%)	9.7	5-11.7	45	1.1	11.6	9.7	80.0	88.9
Volatile Matter (%)	63.0	55.7-67.5	46	1.7	3	60.5	97.8	100
Calorific Value(net) (cal/g)	5317	4767-5857	41	276	5	5861*	85.4	100
Carbon - Total (%)	55.1	46.2-58.6	45	2.0	4	55.9	97.8	100
Carbon - Organic (%)	53.6	43.6-57.8	45	2.3	4	--	97.8	100
Nitrogen (%)	2.0	1.4-2.5	45	0.22	11	2.2	84.4	86.7
Hydrogen (%)	5.8	5.0-6.9	42	0.34	6	4.9	98	100
Sulphur (%)	0.24	0.11-0.36	44	0.04	17	0.38	50.0	79.6
Arsenic (ppm)	<0.1	<0.1-0.4	44	--	--	--	--	---
Mercury (ppm)	0.07	<0.1-0.24	45	0.05	69	--	20.0	20.0
Calcium (ppm)	3149	2143-7027	45	776	25	--	46.7	68.9
Phosphorus (ppm)	967	385-1219	46	207	21	--	43.5	69.6
Potassium (ppm)	1060	768-1680	46	197	19	--	52.2	58.7
Aluminum (ppm)	8929	5704-12254	46	1[illegible]	18	--	45.7	67.4
Iron (ppm)	2332	1343-5523	46	730	31	--	57.5	68.2
Lead (ppm)	11.4	3-39	41	6.7	59	--	31.8	31.8
Manganese (ppm)	29.2	16-72	44	14.1	48	--	31.8	38.6
Magnesium (ppm)	669	461-1453	46	180	27	--	43.5	52.2
Copper (ppm)	14.8	7-20	46	2.5	17	--	69.6	89.2
Zinc (ppm)	10.8	2-30	46	5.8	54	--	26.1	42.7
pH-H_2O	4.5	4.2-5.6	46	0.27	6	--		
pH-CaCl	3.9	3.5-4.5	46	0.26	7	--		

** NRC values are gross calorific values, uncorrected for N, H, S. All other reported calorific values are net calorific values.*

This suggests that precision decreases when the methods are applied to large sets of samples, and that evaluation of effective determination levels should be based on blind replicate and duplicate sets only. However, it should also be noted that a higher degree of homogeneity can be achieved for internal replicate samples (e.g., 10 homogenized subsamples of a ± 0.5 kg sample) than for bulk blind replicate samples (e.g., 25 to 45 x ± 1.0 kg subsamples of a wet bulk sample).

TABLE 4B. BLIND DUPLICATE TEST RESULTS—1984.

Test	"Required" Practical Determination Level (PDL)	% Pairs acceptable ($\Delta/A \leq 1$)	% Acceptable & marginally acceptable ($\Delta/A \leq 1.5$)	PDL required to render duplicate pairs ±80% acceptable ($\Delta/A \leq 1.5$)	% Pairs acceptable at new level
Cation Exchange Capacity	5 meq/100g	29.0	53.4	28 meq/100g	82.2
Conductivity	3 µmhos/cm	30.2	44.2	22 µmhos/cm	77
Fiber Content	1%	59.1	72.1	11%	77
Moisture Content	2%	100	100	-	-
Bulk Density (dry)	0.02 g/cm^3	86.4	88.7	-	-
Absorptive Capacity	0.4	45.5	63.7	4.7	79.6
Ash Content	0.3%	53.7	70.8	0.7%	80.5
Volatile Matter	3.0%	92.5	92.5	-	-
Calorific Value (net)	150 cal/g	90.5	95.3	-	-
Carbon - Total	2.5%	97.7	100	-	-
Carbon - Organic	2.5%	90.9	93.2	-	-
Nitrogen	0.15%	85.4	87.8	-	-
Hydrogen	0.3%	47.7	50.0	0.7%	84.1
Sulphur	0.1 ppm	95.3	95.3	-	-
Arsenic	0.1 ppm	52.6	57.9	0.3 ppm	94.7
Mercury	0.01 ppm	43.9	43.9	0.05 ppm	82.9
Calcium	250 ppm	60.5	67.5	1200 ppm	79.1
Phosphorus	15 ppm	58.1	74.4	80 ppm	79.1
Potassium	10 ppm	48.8	58.1	300 ppm	79.1
Aluminum	50 ppm	25.6	37.2	1500 ppm	79.1
Iron	50 ppm	44.2	55.8	750 ppm	79.1
Lead	2 ppm	31.0	31.0	14 ppm	79.3
Manganese	2 ppm	53.5	69.8	17 ppm	83.7
Magnesium	15 ppm	74.4	86.0	-	-
Copper	1 ppm	62.8	67.5	2.0 ppm	81.4
Zinc	1.5 ppm	48.8	58.1	9 ppm	81.4

TABLE 4C. BLIND REPLICATE TEST RESULTS—1985.

Peat A (1985)

Test	Mean	Range	n =*	SD	% RSD	% Acceptable ($\Delta/A \leq 1$)	% Acceptable & Marginally Acceptable ($\Delta/A \leq 1.5$)
Cation Exchange Capacity (meq/100g)	190	116–255	24	36	19	33	58
Conductivity (µmhos/cm)	70	51–98	24	12	18	50	75
Fiber Content (%)	62	55–71	24	5	8	79	100
Moisture Content (%)	88	86–89	24	1	1	100	100
Bulk Density (dry) (g/cm^3)	0.13	0.12–0.15	25	0.1	8	96	100
Absorptive Capacity	11.5	10.0–12.8	23	0.9	11	78	100
Ash Content (%)	12.2	10.8–13.7	25	0.9	7	88	96
Volatile Matter (%)	63	60.8–64.7	25	1	2	100	100
Calorific Value(net) (cal/g)	4417	4191–4642	25	120	3	100	100
Carbon – Total (%)	50.0	45.8–53.2	23	1.9	5	100	100
Carbon – Organic (%)	50.5	43.6–55.6	25	2.5	5	92	100
Nitrogen (%)	2.4	2.3–2.7	23	0.1	6	96	100
Hydrogen (%)	5.1	4.1–5.6	25	0.5	10	80	84
Sulphur (%)	0.35	0.2–0.5	25	0.09	22	80	80
Arsenic (ppm)	<0.2	<0.1–0.9	24	0.3	150	88	88
Mercury (ppm)	<0.05	<0.03–0.09	24	0.02	40	29	29
Calcium (ppm)	2460	2300–2600	25	214	9	100	100
Phosphorus (ppm)	1857	1600–2140	25	164	9	64	96
Potassium (ppm)	1556	1300–1600	25	153	10	64	84
Aluminum (ppm)	11220	9900–12600	25	981	9	68	100
Iron (ppm)	2200	1800–2700	25	245	11	76	80
Lead (ppm)	34.6	12–60	25	13.5	39	28	40
Manganese (ppm)	39.6	25–50	24	5.7	14	46	83
Magnesium (ppm)	507	470–550	25	22	4	64	100
Copper (ppm)	10.1	3–15	24	2.8	27	50	71
Zinc (ppm)	66.7	48–80	24	6.7	10	75	83
pH–H_2O	4.9	4.6–5.2	23	0.22	5		
pH–CaCl	3.7	3.5–4.0	24	0.13	3		

** n was <25 when anomalous figures were reported, i.e., figures >2 X SD from the mean were considered errors.*

TABLE 4C. CONTINUED.

	Peat B (1985)						
Test	**Mean**	**Range**	**n =***	**SD**	**% RSD**	**% Acceptable ($\Delta/A \leq 1$)**	**% Acceptable & Marginally Acceptable ($\Delta/A \leq 1.5$)**
Cation Exchange Capacity (meq/100g)	171	109-253	24	42	24	33	38
Conductivity (µmhos/cm)	49	42-55	24	5	9	79	100
Fiber Content (%)	48	44-53	24	2.8	6	100	100
Moisture Content (%)	85	83-87	25	1.1	1	100	100
Bulk Density (dry) (g/cm^3)	0.16	0.14-0.18	25	0.01	6	100	100
Absorptive Capacity	7.4	5.3-9.9	25	1.3	18	52	64
Ash Content (%)	11.4	10.5-12.9	24	0.7	7	92	100
Volatile Matter (%)	62	60-64	24	0.9	4	100	100
Calorific Value(net) (cal/g)	5071	4825-5303	23	117	2	100	100
Carbon - Total (%)	53.8	49.6-57.9	23	2.2	4	100	100
Carbon - Organic (%)	53.0	49.1-57.3	23	2.2	4	100	100
Nitrogen (%)	2.1	1.8-2.3	25	0.14	7	96	100
Hydrogen (%)	5.7	4.6-6.3	24	0.4	7	92	100
Sulphur (%)	0.13	0.04-0.25	25	0.06	46	92	92
Arsenic (ppm)	<0.17	<0.1-0.5	25	0.15	82	92	92
Mercury (ppm)	<0.04	<0.03-0.08	23	0.01	25	87	87
Calcium (ppm)	2058	1900-2200	24	97	5	100	100
Phosphorus (ppm)	887	820-960	24	34	4	100	100
Potassium (ppm)	1029	900-1100	24	75	7	100	100
Aluminum (ppm)	15063	14500-16000	24	385	3	100	100
Iron (ppm)	1808	1700-1900	24	58	3	100	100
Lead (ppm)	4.4	<2-8	25	3.7	84	60	60
Manganese (ppm)	20	18-25	24	1.1	6	96	96
Magnesium (ppm)	521	500-550	23	14	3	100	100
Copper (ppm)	4.7	2-9	24	2.1	45	46	46
Zinc (ppm)	<5.6	<2-8	25	2.5	45	32	32
pH-H_2O	4.6	4.4-4.8	24	0.17	4		
pH-CaCl	3.8	3.6-4.0	25	0.10	3		

** n was <25 when anomalous figures were reported, i.e., figures >2 X SD from the mean were considered errors.*

TABLE 4D. BLIND DUPLICATE TEST RESULTS—1985.

TEST	"Required" Practical Determination Level (PDL)	% Pairs acceptable ($\Delta/A \leq 1$)	% Acceptable & marginally acceptable ($\Delta/A \leq 1.5$)	PDL required to render duplicate pairs ±80% acceptable ($\Delta/A \leq 1.5$)	% Pairs acceptable at new level
Cation Exchange Capacity	5 meq/100g	28.6	38.1	69 meq/100g	81
Conductivity	3 µmhos/cm	52.4	66.7	5 µmhos/cm	86
Fiber Content	1%	71.4	81%	< 1.0%*	81
Moisture Content	2%	100	100	< 0.5%*	100
Bulk Density (dry)	0.02 g/cm³	95.2	95.2	< 0.01 g/cm³*	95
Absorptive Capacity	0.4	66.7	81.0	< 0.4*	81
Ash Content	0.3%	85	95	< 0.3%*	95
Volatile Matter	3.0%	100	100	< 2.0%	100
Calorific Value (net)	150 cal/g	90.5	100	<125 cal/g*	100
Carbon - Total	2.5%	100	100	< 2.0%*	100
Carbon - Organic	2.5%	100	100	<2.0%*	100
Nitrogen	0.15%	95.2	95.2	< 0.15%*	100
Hydrogen	0.3%	90.5	100	< 0.3%*	100
Sulphur	0.1%	100	100	0.05%	81
Arsenic	0.1 ppm	90.5	100	< 0.1 ppm*	100
Mercury	0.01 ppm	85.7	85.7	0.01 ppm	86
Calcium	250 ppm	80	85	<250 ppm*	85
Phosphorus	15 ppm	80	95	<15 ppm*	95
Potassium	10 ppm	60	65	68 ppm	85
Aluminum	50 ppm	85	85	<50 ppm*	85
Iron	50 ppm	85	95	<50 ppm*	95
Lead	2 ppm	90	90	< 2 ppm*	90
Manganese	2 ppm	85	90	< 2 ppm	90
Magnesium	15 ppm	90	95	<15 ppm*	95
Copper	1 ppm	70	70	3 ppm	90
Zinc	1.5 ppm	75	75	1.5 ppm	75

** PDL for method is lower than required in the column at the left, but is not directly calculable because the mean of each duplicate pair is more than 10X the required PDL. As a result, the factor of 1.5 X $\bar{x}$ applies independent of how much lower the PDL is reduced. Samples with lower levels of some of these, i.e., Al and Fe, would enable better quantification of the actual PDLs achieved.*

4.4.1 RESULTS

a) Cation Exchange Capacity

*52C-37 B1500E	C1	Mean	133.7 meq/100g
		SD	12.7
		RSD	9.5%
*52G-155 L500W+200S	C1	Mean	136.0 meq/100g
		SD	14.6
		RSD	10.7%

b) pH (H_2O)

°*52C-37 B1500E	C1	Mean	4.0
		SD	.0
		RSD	0%
°*52G-155 L500W +200S	C1	Mean	3.95
		SD	0.05
		RSD	1.3%

c) pH ($CaCl_2$)

°*52C-37 B1500E	C1	Mean	3.18
		SD	0.04
		RSD	1.3%
°52G-155 L500W+200S	C1	Mean	2.68
		SD	0.1
		RSD	3.7%

d) Conductivity

52C-37 B1500E	C1	Mean	16.1 µmhos/cm
		SD	3.93
		RSD	24.4%
*52G-155 L500W+200S	C1	Mean	21.8 µmhos/cm
		SD	1.93
		RSD	8.9%

e) Fiber

*52C-37 B1500E	C1	Mean	94.5%
		SD	7.5
		RSD	7.9%
52G-155 L500W+200S	C1	Mean	88.8%
		SD	3.7
		RSD	4.2%

f) Moisture Content

*31M-21 H1300N	C1	Mean	92.2%
		SD	0.17
		RSD	0.2%
*52G-155 L500W+200S	C1	Mean	94.2%
		SD	0.16
		RSD	0.2%

g) Bulk Density (dry)

*52C-37 B1500E	C1	Mean	0.06 g/cm^3
		SD	0.003
		RSD	5.2%
52C-32 B3500S	C2	Mean	0.07 g/cm^3
		SD	0.007
		RSD	9.2%

h) Absorptive Capacity

*52C-37 B1500E	C1	Mean	32.3
		SD	1.2
		RSD	3.7%
52C-32 B3500S	C2	Mean	17.4
		SD	2.8
		RSD	16.1%

i) Ash Content

*52C-37 B5700E	C1	Mean	8.3%
		SD	0.3
		RSD	3.6%
52G-165 L000N+700W	C5	Mean	3.5%
		SD	0.3
		RSD	8.1%

j) Volatile Matter

*31B-8 L3500N+300E	C6	Mean	68.8%
		SD	0.3
		RSD	0.4%
*52C-37 B5700E	C1	Mean	67.3%
		SD	0.3
		RSD	0.5%

k) Calorific Value

*31C-569 F900N	C3	Mean	4444 cal/g
		SD	32
		RSD	0.7%
*52G-191A B100E	C5	Mean	5099 cal/g
		SD	43
		RSD	0.9%

l) Total Carbon

*31B-18 L1200W+400S	C3	Mean	45.3%
		SD	0.6
		RSD	1.4%
*52C-44 B4900S	C4	Mean	51.9%
		SD	0.4
		RSD	0.7%

m) Organic Carbon

31C-569 H1100N	C5	Mean	37.7%
		SD	1.1
		RSD	3.1%
52G-181 G2500S+100W	C6	Mean	40.7%
		SD	2.1
		RSD	5.2%

n) Nitrogen

*31C-569 H1100N	C1	Mean	3.5%
		SD	0.1
		RSD	2.4%
*52G-119 G800E+500N	C1	Mean	2.3%
		SD	0.1
		RSD	3.0%

o) Hydrogen

*31E-348 F800N	C4	Mean	1.9%
		SD	0.1
		RSD	4.8%
*52G-190 F2200N	C2	Mean	1.7%
		SD	0.1
		RSD	7.6%

p) Sulphur

°31E-348 F800N	C4	Mean	0.1%
		SD	0.03
		RSD	24.2%
°52G-190 F2200N	C2	Mean	0.1%
		SD	0.01
		RSD	9.1%

q) Mercury

52C-37 B1500E	C1	Mean	0.07 ppm
		SD	0.01
		RSD	17.3%
52G-155 L500W+200S	C1	Mean	0.04 ppm
		SD	0.01
		RSD	21.5%

r) Arsenic

31C-521 B300W	C7	Mean	5.0 ppm
		SD	0.5
		RSD	10.0%
52D-16 B1500N	C3	Mean	4.0 ppm
		SD	1.0
		RSD	24.9%

s) Calcium

31M-21 F300N	C2	Mean	12342 ppm
		SD	2034
		RSD	16.5%
*52C-37 B5700E	C1	Mean	3746 ppm
		SD	90
		RSD	2.4%

t) Phosphorus

*31M-21 F300N	C2	Mean	333 ppm
		SD	15
		RSD	4.6%
*52C-37 B5700E	C1	Mean	789 ppm
		SD	16
		RSD	2.0%

u) Potassium

*31M-21 F300N	C2	Mean	1102 ppm
		SD	89
		RSD	8.1%
*53C-37 B5700E	C1	Mean	2886 ppm
		SD	174
		RSD	6.0%

v) Aluminum

31M-21 F300N	C2	Mean	2764 ppm
		SD	397
		RSD	14.4%
*52C-37 B5700E	C1	Mean	6176 ppm
		SD	120
		RSD	1.9%

w) Iron

31M-21 F300N	C2	Mean	2117 ppm
		SD	405
		RSD	19.1%
*52C-37 B5700E	C1	Mean	5957 ppm
		SD	123
		RSD	2.1%

x) Lead

°31M-21 F300N	C2	Mean	3.3 ppm
		SD	1.3
		RSD	37.9%
52C-37 B5700E	C1	Mean	14.1 ppm
		SD	4.6
		RSD	32.7%

y) Manganese

*31M-21 F300N	C2	Mean	50.3 ppm
		SD	5.2
		RSD	6.4%
*52C-37 B5700E	C1	Mean	119.8 ppm
		SD	2.0
		RSD	1.7%

z) Magnesium

*31M-21 F300N	C2	Mean	1465 ppm
		SD	111
		RSD	7.6%
*52C-37 B5700E	C1	Mean	1330 ppm
		SD	26
		RSD	2.0%

aa) Copper

*31M-21 F300N	C2	Mean	16.6 ppm
		SD	1.0
		RSD	5.8%
52C-37 B5700E	C1	Mean	22.7 ppm
		SD	4.8
		RSD	20.1%

bb) Zinc

*31M-21 F300N	C2	Mean	8.9 ppm
		SD	0.6
		RSD	6.4%
*52C-37 B5700E	C1	Mean	47.2 ppm
		SD	1.1
		RSD	2.4%

4.4.2 1985 Results

a) Cation Exchange Capacity

* 52K-85 L1900S+200E	C3	Mean	120.6 meq/100g
		SD	3.1
		RSD	2.6%
52J-29 B1000N	C1	Mean	231.6 meq/100g
		SD	31.2
		RSD	14.3%

b) pH (H_2O)

° 52J-85 L1900S+200E	C2	Mean	4.3
		SD	0.16
		RSD	3.6%
° 52J-29 L1900N+400E	C1	Mean	5.3
		SD	0.25
		RSD	4.8%

c) pH ($CaCl_2$)

° 52K-85 L1900S+200E	C2	Mean	3.3
		SD	0.07
		RSD	2.0%
° 42A-24 B1500S	C1	Mean	3.9
		SD	0.16
		RSD	4.2%

d) Conductivity

52K-85 L1900S+200E	C2	Mean	58.8 µmhos/cm
		SD	5.4
		RSD	9.1%
52J-29 L1900N+400E	C1	Mean	95.0 µmhos/cm
		SD	16.5
		RSD	17.4%

e) Fiber

* 52K-85 L1900S+200E	C2	Mean	54.2%
		SD	1.8
		RSD	3.4%
42A-24 L2000S+100W	C1	Mean	97.9%
		SD	3.8
		RSD	3.8%

f) Moisture Content

* 52K-85 B700S	C5	Mean	89.7%
		SD	0.44
		RSD	0.5%
* 52J-29 B400N	C2	Mean	88.3%
		SD	0.26
		RSD	0.3%

g) Bulk Density (dry)

52K-86 B300S	C1	Mean	0.08 g/cm^3
		SD	0.005
		RSD	6.5%
* 52J-29 L1900N+400E	C3	Mean	0.11 g/cm^3
		SD	0
		RSD	0%

h) Absorptive Capacity

* 52K-86 B300S	C1	Mean	27.0
		SD	0.0
		RSD	3.7%
* 52J-29 L1900N+400E	C3	Mean	13.0
		SD	0.6
		RSD	4.6%

i) Ash Content

52J-113 B200N	C1	Mean	3.6%
		SD	0.39
		RSD	10.9%
* 52J-113 L700N+300W	C1	Mean	8.6%
		SD	0.22
		RSD	2.5%

j) Volatile Matter

52F-60 L2500N+200E	C5	Mean	70.6%
		SD	2.8
		RSD	4.0%
* 42A-24 L2000S+100W	C5	Mean	63.3%
		SD	0.4
		RSD	0.7%

k) Calorific Value

* 52J-185 B900E	C2	Mean	4957 cal/g
		SD	16.3
		RSD	0.3%
* 42A-171 L3600E +600N	C1	Mean	4339 cal/g
		SD	44.7
		RSD	1.0%

l) Total Carbon

* 52F-60 B1500N	C1	Mean	45.7%
		SD	0.3
		RSD	0.6%
* 42A-24 L2000S+100W	C5	Mean	51.2%
		SD	0.3
		RSD	0.5%

m) Organic Carbon

* 52F-60 B1500N	C1	Mean	43.6%
		SD	0.3
		RSD	0.7%
* 42A-24 L2000S+100W	C5	Mean	50.4%
		SD	0.3
		RSD	0.6%

n) Nitrogen

52K-20 L500E+600N	C3	Mean	1.5%
		SD	0.1
		RSD	3.6%
52J-113 B200N	C4	Mean	1.3%
		SD	0.2
		RSD	17.8%

o) Hydrogen

* 52F-60 B1500N	C1	Mean	4.9%
		SD	0.1
		RSD	1.3%
* 42A-24 L2000S+100W	C5	Mean	5.7%
		SD	0.1
		RSD	1.4%

p) Sulphur

° 52F-60 B1500N	C1	Mean	0.02%
		SD	0.01
		RSD	47.0%
° 42A-24 L2000S+100W	C5	Mean	0.07%
		SD	0.02
		RSD	23.4%

q) Mercury — No data.

r) Arsenic

°* 52F-57 B300N	C3	Mean	<0.1 ppm
		SD	0
		RSD	0%
* 42L-264 B1400E	C1	Mean	4.4 ppm
		SD	0.3
		RSD	6.5%

s) Calcium

* 52J-113 B200N	C1	Mean	5808 ppm
		SD	172
		RSD	3.0%
* 52J-113 L700N+300W	C1	Mean	13887 ppm
		SD	219
		RSD	1.6%

t) Phosphorus

* 52J-113 B200N	C1	Mean	381 ppm
		SD	12
		RSD	3.1%
* 52J-113 L700N+300W	C1	Mean	698 ppm
		SD	12
		RSD	1.7%

u) Potassium

* 52J-113 B200N	C1	Mean	1439 ppm
		SD	34
		RSD	2.3%
52J-113 L700N+300W	C1	Mean	1264 ppm
		SD	69
		RSD	5.5%

v) Aluminum

* 52J-113 B200N	C1	Mean	986 ppm
		SD	25
		RSD	2.5%
* 52J-113 L700N+300W	C1	Mean	3136 ppm
		SD	79
		RSD	2.5%

w) Iron

52J-113 B200N	C1	Mean	626 ppm
		SD	19
		RSD	3.0%
52J-113 L700N+300W	C1	Mean	2216 ppm
		SD	66
		RSD	3.0%

x) Lead

* 52J-113 B200N	C1	Mean	13.6 ppm
		SD	3.1
		RSD	22.4%
* 52J-113 L700N+300W	C1	Mean	18 ppm
		SD	0
		RSD	0%

y) Manganese

* 52J-113 B200N	C1	Mean	30 ppm
		SD	1.8
		RSD	2.2%
°* 52J-113 L700N-300W	C1	Mean	30 ppm
		SD	0.9
		RSD	2.9%

z) Magnesium

* 52J-113 B200N	C1	Mean	1108 ppm
		SD	26
		RSD	2.4%
* 52J-113 L700N+300W	C1	Mean	2130 ppm
		SD	19
		RSD	0.9%

aa) Copper

° 52J-113 B200N	C1	Mean	1.9 ppm
		SD	0.6
		RSD	29.8%
* 52J-113 L700N+300W	C1	Mean	3.6 ppm
		SD	0
		RSD	0%

bb) Zinc

52J-113 B200N	C1	Mean	28.1 ppm
		SD	1.4
		RSD	4.9%
52J-113 L700N+300W	C1	Mean	25.5 ppm
		SD	3.1
		RSD	12.3%

5.0 Standards and Blanks

For parameters not reported here, about 10 percent of samples were repeated as checks on drift and repeatability, and some samples were used as standards to be rechecked following set lengths of sample runs.

5.1 1984 RESULTS

5.1.1 ASH CONTENT

Oven-dry, milled Peat A (1984) was run after every 20 samples.

Mean	11.15%
SD	1.6
RSD	14.4%
n = 47	

The RSD for Peat A (1984) (n = 28) run as blind replicates was 9.6 percent (mean = 12.3% ash content). National Research Council (NRC) reported a mean of 13.1 percent ash content. No drift was apparent through the course of analysis.

5.1.2 VOLATILE MATTER

Oven-dry, milled Peat A (1984) was run after every 25 samples.

Mean	66.26%
SD	1.6
RSD	3.7%
n = 40	

The RSD for Peat A (1984) (n = 29) run as blind replicates was 2 percent (mean = 65.3% volatile matter). NRC reported a mean of 62.1 percent. No drift was apparent through the course of analysis.

5.1.3 CALORIFIC VALUE

1. Benzoic acid was run periodically as a standard of known value, and also as the standard for the temperature rise in the calorimeter.

Mean	2403 cal/°C
SD	90.4
RSD	3.8%
n = 40	

This was based on a mean value for benzoic acid run as a daily standard of 6318 cal/g (recommended value 6310 cal/g).

2. Oven-dry, milled Peat A (1984) was run after approximately every 20 samples.

Mean	4304 cal/g (uncorrected for N, H, S)
SD	45
RSD	1.1%
n = 47	

The RSD for Peat A (1984) (n = 20) run as blind replicates in 1984 was 5 percent (mean = 4536 cal/g, uncorrected for N, H, S). This difference of 232 cal/g is inexplicable, unless there was inhomogeneity between the Peat A (1984) used as dry bulk sample and that used for wet bulk samples. Refer to 1985 results with same batch of Peat A (1984) as a standard (Section 5.2.3).

5.1.4 TOTAL CARBON

Oven-dry, milled Peat A (1984) was run after approximately every 20 samples.

Mean	47.5%
SD	0.4
RSD	0.9%
n = 49	

The RSD for Peat A (1984) run as blind replicates (n = 32) was 4 percent (mean = 47.1% total carbon). NRC reported a mean of 47.5 percent total carbon. No drift in values was apparent.

Daily calibrations were run on carbon, including blanks. All blanks run were less than 0.1 percent. Carbon reburn values were run on 5 samples. All reburns were less than 0.1 percent.

5.1.5 NITROGEN

1. National Bureau of Standards (NBS) 1571 was run after approximately every 60 samples and NBS 1575 was run after approximately every 40 samples.

NBS 1571	Rec. Value	2.76%
	Mean	2.59%
	SD	0.62
	RSD	23.9%
	n = 17	
	% of Rec. Value	93.8%
NBS 1575	Rec. Value	1.2%
	Mean	1.30%
	SD	0.35
	RSD	26.9%
	n = 23	
	% of Rec. Value	108.3%

2. Equipment controls.

$(NH_4)_2SO_4$ at 9.9%	
Mean	9.87%
SD	0.05
RSD	0.5%
n = 10	
$(NH_4)_2SO_4$ at 0.3136%	
Mean	0.3152%
SD	0.005
RSD	1.6%
n = 10	

3. Blanks were used daily to zero equipment.

5.1.6 HYDROGEN

The following inorganic hydrogen standards were run in 1984. (All analyses were rerun in 1985 because reported peat values were too low despite

these standards. In 1984, H and S were done in the same burn; in 1985, all samples and reruns of 1984 samples had separate burns for H and S.)

1. Four standards were run to provide internal quality control, interspersed in the sample run.

BX-N (Association Nationale de la Recherche Technique, France)

Rec. Value	11.69% (bauxite)
Test Mean	11.71%
SD	0.3
RSD	2.9%

n = 11

GL-O (Association Nationale de la Recherche Technique, France)

Rec. Value	5.72% (glauconite)
Test Mean	5.78%
SD	0.23
RSD	4.0%

n = 15

UB-N (Association Nationale de la Recherche Technique, France)

Rec. Value	11.28% (serpentine)
Test Mean	11.13%
SD	0.29
RSD	2.6%

n = 9

NIM-L (National Institute for Metallurgy, South Africa)

Rec. Value	2.31% (lujavrite)
Test Mean	2.40%
SD	0.16
RSD	6.7%

n = 12

2. Blanks

Mean	0.0054%
SD	0.0029

n = 98

5.1.7 SULPHUR

NBS 1571 (orchard leaves) were run after every 20 samples as standards of known value.

Rec. Value	0.19%
Test Mean	0.177%
SD	0.036
RSD	20.3%

n = 47

% of Rec. Value 93%

The RSDs for test peats run as blind replicates were 14 percent (mean = 0.6%, n = 33) and 17 percent (mean = 0.82%, n = 44).

5.1.8 MERCURY

1. Four standards were interspersed in the sample run. Test runs did not come close enough to recommended values, and indicate a PDL for this method higher than 0.02 ppm.

NBS 1575 (pine needles)

Rec. Value	0.155 ppm
Test Mean	0.092
SD	0.017
RSD	18.5%

n = 9

% of Rec. Value 59.5%

SO-1 (CCRMP (Canadian Certified Reference Materials Project, CANMET), Canada)

Rec. Values	0.022 ppm
Test Mean	0.011
SD	0.004
RSD	36.3%

n = 5

% of Rec. Value 50%

SO-2 (CCRMP, Canada)

Rec. Values	0.082 ppm
Test Mean	0.032
SD	n.a.

n = 4

% of Rec. Value 39.0%

NBS 1573 (tomato leaves)

Rec. Values	0.1 ppm
Test Mean	0.063
SD	0.021
RSD	33.3%

n = 33

% of Rec. Value 63%

2. Blanks were run to assess the need for any compensations attributed to the method.

Mean <0.01 ppm

n = 18

5.1.9 ARSENIC

1. Two standards were run to provide internal quality control, interspersed in the sample run.

SY2 (syenite)

Rec. Value	18 ppm
Test Mean	19.8
SD	1.51
RSD	7.6%

n = 23

% of Rec. Value 110%

NBS 1571 (orchard leaves)

Rec. Value	10 ppm
Test Mean	9.77
SD	1.18
RSD	12.1%

n = 14

% of Rec. Value 97.7%

2. Blanks were run to assess the need for any compensations attributable to the method.

Mean = <0.13 ppm (<0.01 - 0.03).

n = 34

5.1.10 CATION EXCHANGE CAPACITY

1. $(NH_4)_2SO_4$ standards at 0.015 percent N and 0.3 percent N were run as blind replicates.

Rec. Value	0.15%N	Rec. Value	0.3%N
Test Mean	0.159%	Test Mean	0.314%
SD	0.0008	SD	0.0043
RSD	0.5%	RSD	1.37%
n = 27		n = 31	
% of Rec. Value	106%	% of Rec. Value	105%

2. Blanks

Mean	0.0019%N
SD	0.001
n = 29	

5.1.11 MULTIELEMENT ICAP ANALYSIS

The peat samples were run in batches of approximately 120 samples. The beginning of each batch started with:

blank;

blank;

Ca interference correction solution;

Fe interference correction solution;

Al interference correction solution;

blank;

blank;

control solution.

Then, the samples were run in sets of:

10 samples;

NBS 1571, 1575, Peat A, or Peat B;

control solution;

blank.

Every 40 samples, another set of solutions identical with the starting set was run. Accordingly, for a batch of 120 samples, approximately 190 solutions were run. All samples were made up with 1 ppm Be as an internal standard.

For the first batch of samples run, a control solution was made by mixing a few millilitres of each of the first 40 samples. After completion of the first batch (120 samples), the entire remaining solution of all the first batch of samples was mixed into a single solution and used for the rest of the analysis. This source of machine control solution related to the ICAP conditions in the same manner used as the peat samples, allowing for very accurate monitoring of the ICAP.

Corrections were then applied as follows.

1. Fe interference on Be was corrected.
2. All elements except Pb were normalized to a fixed Be count. (Pb was found not to relate to Be as an internal standard.)
3. Blank corrections: blank values were evaluated by a chemist to observe if there was any drift, level shift, oscillations, irregular jumps, or contamination. For some batches, a single blank value would apply to the entire batch. However, if drift was noticeable, or if shifts were noticeable, then the blank correction was done on groups of 10 or 20 samples. Irregular blanks and those appearing to be contaminated were not used in these corrections.
4. Using the Ca, Fe, and Al interference solutions (solutions high in those elements but with no detectable amounts of the other elements being analyzed), the sample values were interference corrected. These interference solutions were spectroscopic standard solutions (SPEX). For some batches, a single factor value was used for the entire batch; for other batches, the factor changed between groups of 40 samples, and were accordingly applied as necessary to the samples.
5. The results were scaled so that the control solution values were constant. As with the blanks and interference corrections, this was done either as a general scaling, or on groups of 10 or 20 samples, as appropriate.

A. Four standards were run to provide internal quality control. The results (Table 5) should be assessed in relation to the values achieved in blind replicate tests and the tests of the acid dissolution methods.

B. An assessment of possible uncompensated drift in the reported values of 31 control samples of NBS 1571, 29 NBS 1575, 26 Peat A (1984), and 28 Peat B (1984) was made in relation to the "effective" Practical Detection Limits (PDL) suggested by duplicate and replicate tests. Only the Zn values for NBS 1575 showed apparent drift of more than 10 percent at values greater than 10 x PDL.

5.2 1985 RESULTS

5.2.1 ASH CONTENT

1. An oven-dry, internal peat sample (TSL-1), without known recommended values, was run after about every 20 samples for internal lab control.

Mean	3.5%
SD	0.19
RSD	5.3%
n = 37	

No drift was apparent through the course of analysis.

2. Oven-dry milled Peat A (1984) was run after every 20 samples.

Mean	12.4%
SD	0.4%
RSD	3.3%
n = 35	

TABLE 5. 1984 RESULTS OF STANDARDS AND CONTROLS RUN IN MULTIELEMENT ICAP ANALYSIS (ppm).

Elements (ppm)	Al	Fe	Ca	Mg	K	Mn	P	Cu	Pb	Zn
A. NBS 1571 (orchard leaves)										
Rec. Value	---	300	20,900	6,200	14,700	91	2,100	12	45	25
Mean of Standard Tests (n = 31)	490	323	18,761	6,504	15,218	85	2,130	12	42	24
SD	139	55	1,401	610	1,391	4.7	115	2.6	9.8	4.2
% RSD	28	17	7.5	9.4	9.1	5.5	5.4	22	23	18
% of Rec. Value	---	108	90	105	104	93	101	100	93	96
B. NBS 1575 (pine needles)										
Rec. Value	545	200	4,100	--	3,700	675	1,200	3	11	--
Mean of Standard Tests (n = 29)	557	227	4,254	1,396	3,930	668	1,285	3.3	14	69
SD	56	100	276	88	664	39	85	1.6	5	12.5
% RSD	10	44	6.5	6.3	17	5.8	6.6	48	35	18
% of Rec. Value	102	114	104	---	106	99	107	110	127	--
C. Peat A (1984) (dried, milled homogenized peat)										
Values from blind Replicate Tests (n = 31)	6,604	2,454	3,165	747	1,738	38	1,367	20	68.1	91.5
% RSD	20	24	27	24	20	17	11	22	18	23
Mean of Standard Tests (n = 26)	6,844	2,260	2,609	674	1,589	36	1,604	21	75	88
SD	1,153	522	231	49	177	2.1	134	7.9	14	15
% RSD	17	23	8.9	7.3	11	5.8	8.4	38	19	17
% of Values from Blind Replicate Tests	104	92	82	90	91	95	118	105	110	96
D. Peat B (1984)										
*Values from Blind Replicate Tests (n = 46)	8,892	2,332	3,149	669	1,060	29.2	967	14.8	11.4	10.8
% RSD	18	32	25	27	19	48	21	17	59	54
Mean of Standard Tests (n = 28)	7,285	4,118	4,986	1,083	1,441	78	411	23	14	14
SD	1,860	472	1,417	110	256	6	42	7.3	5.3	6.7
% RSD	25	12	28	10	18	8	10	32	38	48
% of Values from Blind Replicate Tests	81	177	158	162	136	267	43	155	123	130

** Blind replicate peats may be more heterogeneous because replicates are individually dried and milled, whereas test peats were subsampled following drying and milling.*

The RSD for Peat A (1984) (n = 28) run as blind replicates in 1984 was 9.6 percent (mean = 12.3% ash content). No drift was apparent through the course of analysis.

5.2.2 VOLATILE MATTER

1. Oven-dry, milled Peat A (1984) was run after approximately every 20 samples.

Mean	64.3%
SD	1.8
RSD	2.8%

n = 40

The RSD for Peat A (1984) (n = 29) run as blind replicates in 1984 was was 2 percent (mean = 65.3% volatile matter). NRC reported a mean of 62.1 percent. No drift was apparent through the course of analysis.

2. Oven-dry milled Peat B (1984) was run as a control early in the run (n = 5).

Mean	64.9%
SD	0.7
RSD	1.0%

The RSD for Peat B (1984) (n = 46) run as blind replicates in 1984 was 3 percent (mean = 63.0% volatile matter). NRC reported a mean of 60.5 percent.

3. A Leco coke standard (501-531, Lot 884) was run as a standard.

Mean	39.9%
SD	0.8
RSD	2.1%

n = 5

Recommended value = 39.0%

5.2.3 CALORIFIC VALUE

1. Benzoic acid was run as a standard after approximately every 20 samples.

Mean	6319 cal/ (uncorrected for N, H, S)
SD	78.8
RSD	1.3%

n = 39

Recommended value 6310 cal/g. No drift was apparent through the course of analysis.

2. Oven-dry, milled Peat A (1984) was run as a control.

Mean	4364 cal/g (uncorrected for N, H, S)
SD	65
RSD	1.5%

n = 8

The RSD for Peat A (1984) run as blind replicates in analyses redone in 1985 (n = 20) was 5 percent (mean = 4536 cal/g, uncorrected for N, H, S). This difference of 172 cal/g exactly parallels the 1984 control results (Section 5.1.3).

3. Oven-dry, milled Peat B (1984) was run occasionally as a control.

Mean	5167 cal/g (uncorrected for N, H, S)
SD	227
RSD	4.4%

n = 7

The RSD for Peat B (1984) run as blind replicate in analyses redone in 1985 (n = 20) was 5 percent (mean = 5457 cal/g, uncorrected for N, H, S). This difference of 290 cal/g, and the difference noted above with Peat A (1984), is possibly attributable to sample heterogeneity (e.g., the oven-dry peat used as a control came from a single subsample of the bulk replicate material, but the data on blind replicates was derived from all of the subsamples).

5.2.4 TOTAL CARBON

Oven-dry, milled Peat A (1984) was run after approximately every 30 samples.

Mean	47.4%
SD	0.4
RSD	8.7%

n = 22

The RSD for Peat A (1984) run as blind replicates (n = 32) in 1984 was 4 percent (mean = 47.1% total carbon). NRC reported a mean of 47.5 percent total carbon. No drift in values was apparent.

5.2.5 NITROGEN

1. NBS 1575 (pine needles) were run as a standard of known value.

Mean	1.3%
SD	0.17
RSD	13.3%

n = 3

Recommended value = 1.2%

2. NBS 1573 (tomato leaves) were run as a standard of known value.

Mean	4.6%
SD	0.1
RSD	2.2%

n = 3

Recommended value = 5.0%

3. A Leco coke standard (501-531, Lot 884) was run as a standard of known value.

Mean	1.25%

n = 2

Recommended value = 1.35%

4. Oven-dry, milled Peat A (1984) was run as a control.

Mean	2.18%
SD	0.09
RSD	4.1%

n = 5

The RSD for Peat A (1984) run as blind replicates (n = 30) in 1984 was 18 percent (mean = 2.3%). NRC reported a mean of 2.1 percent N.

5. Oven-dry, milled Peat B (1984) was run as a control.

Mean	2.34%
SD	0.17
RSD	7.3%

n = 7

The RSD for Peat B (1984) run as blind replicates (n = 45) in 1984 was 11 percent (mean = 2.0%). NRC reported a mean of 2.2 percent N.

6. Repeats done on samples (n = 65) showed no apparent drift through the course of analysis.

5.2.6 HYDROGEN

1. Benzoic acid was run as a standard of known value after approximately every 12 samples. No drift was apparent.

Mean	4.95%
SD	0.08
RSD	1.6%

n = 57

Recommended value = 4.95%

2. A Leco coke standard (501-531, Lot 884) was run as a standard after approximately every 50 samples. No drift was apparent.

Mean	4.90%
SD	0.07
RSD	1.4%

n = 11

Recommended value = 4.90%

5.2.7 SULPHUR

Oven-dry, milled Peat A (1984) was run after approximately every 35 samples. No drift was apparent in the course of analyses.

Mean	0.55%
SD	0.025
RSD	4.5%

n = 19

The RSD for Peat A (1984) run in 1984 as blind replicate (n = 33) was 14 percent (mean = 0.6% sulphur).

5.2.8 MERCURY

1. Six standards were interspersed in the sample run.

NBS 1575 (pine needles)

Rec. Value	0.15 ppm
Test Mean	0.126
SD	0.015
RSD	12%

n = 9

% of Rec. Value 84%

NBS 1573 (tomato leaves)

Rec. Value	0.1 ppm
Test Mean	0.09
SD	0.012
RSD	14%

n = 7

% of Rec. Value 85%

SO-1 (CCRMP, Canada)

Rec. Value	0.022 ppm
Test Value	<0.03

n = 6

SO-2 (CCRMP, Canada)

Rec. Value	0.082 ppm
Test Value	0.072
SD	0.015
RSD	20%

n = 6

% of Rec. Value 88%

Peat A (1984)

1984 blind replicate mean	0.13 ppm (n = 34)
Test Value	0.09
SD	0.015
RSD	17%

n = 9

Peat B (1984)

1984 blind replicate mean	0.07 ppm (n = 45)
Test Value	0.075
SD	0.01
RSD	13%

n = 2

2. Blanks were run to assess the need for any compensation attributable to the method.

Mean <0.03 ppm
n = 7

5.2.9 ARSENIC

1. Six standards were interspersed in the sample run.

SY2 (syenite)

Rec. Value	18 ppm
Test Mean	13.2
SD	1.7
RSD	13%

n = 6

% of Rec. Value 73%

SY3 (syenite)

Rec. Value	20 ppm
Test Mean	16.9
SD	1.7
RSD	11%

n = 5

% of Rec. Value 85%

NBS 1575 (pine needles)

Rec. Value	0.21 ppm
Test Mean	<0.1

n = 4

NBS 1573 (tomato leaves)

Rec. Value	0.27 ppm
Test Value	<0.1

n = 5

Peat A (1984)

1984 blind replicate mean	<0.81 ppm (n = 31)
Test Value	<0.4

n = 4

Peat B (1984)

1984 blind replicate mean	<0.1 ppm (n = 44)
Test Value	<0.1

n = 6

2. Blanks were run to assess the need for any compensation attributable to the method.

Mean <0.1 ppm
n = 8

5.2.10 CATION EXCHANGE CAPACITY

Blanks were run to assess the need for any compensation attributable to the method.

Mean <0.0009%N
SD <0.0010
n = 21

5.2.11 MULTIELEMENT ICAP ANALYSIS

The peat samples were run in batches of 40, made up of 4 groups of 10 samples, in the following order:

blank (used as zeroing point for first batch of 10);

rock standard control (blind);

rock standard control (same as above, blind);

blank (same as above, to check for drift);

blank (used as zeroing point for second batch of 10);

SY-2 sample;

10 samples;

SY-2 sample (same as above);

blank (same as that above for zeroing second batch of 10);

repeated twice for batch of 40 samples.

For the ICAP "whole rock procedure", the reference standard materials used as controls were run as blind controls with every batch of samples, and each batch was accepted or re-run conditional on the ICAP results of the blind reference standard controls being acceptable or not. This procedure used the blind standards to ensure accuracy as well as precision. Because no scaling or normalization occurred in this technique, the actual order of standards was not relevant to the results. As well, because each blank was used as a zeroing value and as a check on drift, all blank values were the same (i.e., 0).

A. Organic Standard

Five blind replicate samples of Peat A (1984) were part of the main run of samples (Table 6a).

B. Whole Rock Standards

Peat A (1984) shown in Table 6a has an ash content of about 12.3 percent. During ICAP analysis, the peat sample is rendered 95 to 100 percent inorganic ash, and run in a manner more or less comparable to whole rock analyses. Therefore, the values of Peat A (1984) as recorded by the ICAP (concentrated as ash) are in the order of (100/12.3)X the above values:

	Al	Fe	Ca	Mg	K	Mn	P	Zn
ppm	96057	16480	18504	3862	17797	309	15522	642

Expressed as percent oxides, these values correspond to:

	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	MnO	P_2O_5
%	18.154	2.123	2.589	0.647	2.144	0.04	3.556

The ICAP's matrix effects in the whole rock analyses may differ from those in peat analyses, but of the 20 whole rock standards used, the results shown in Table 6b may be particularly relevant to these tests.

The accuracy and precision of these results is so much better than obtained on peat (or organic standards) in 1984 or 1985, that it is worth noting that relative standard deviations obtained on these controls should not be expected to be achieved on peat samples, which are simply too heterogenous to produce comparable precision. However, these whole rock standards confirm the accuracy and precision of the method and equipment. It is also worth cautioning that, if organic standards or controls are used in this method of ICAP sample preparation, a laboratory should be prepared to commit a large volume of very well mixed material to satisfy the requirement of 10 g per sample run.

5.2.12 LEAD

MESS-1 (NRC, Canada)

Rec. Value	34 ppm
Test Value	20
SD	7.1
RSD	36%
n = 15	
% of Rec. Value	59%

BCSS (NRC, Canada)

Rec. Value	23 ppm
Test Value	13
SD	5.3
RSD	41%
n = 16	
% of Rec. Value	57%

In both these runs of standards, values increased towards the end of the runs. In the case of sample BCSS, the mean of values in the last third of the run was more than double the mean of values in the first third of the run.

TABLE 6A. 1985 RESULTS OF CONTROLS RUN IN MULTIELEMENT ICAP ANALYSIS (ppm).

Peat A (1984) (dried, milled homogenized peat)

	Al	Fe	Ca	Mg	K	Mn	P	Zn
Test Value (n = 5)	11,815	2,027	2,276	475	1,451	38	1,909	79
SD	216	103	49	9	73	1.5	30	3
RSD	2%	5%	2%	2%	5%	4%	2%	3%
1984 mean of standards test (n = 26)	6,844	2,260	2,609	674	1,589	36	1,604	88
1984 mean of blind replicate tests (n = 30,31)	6,604	2,454	3,165	747	1,738	38	1,367	92
% of 1984 mean of standards tests	173%	90%	87%	71%	91%	106%	119%	90%

Blanks were run after every 10 samples. All values were less than 1 ppm.

5.2.13 COPPER

MESS-1 (NRC, Canada)

Rec. Value	25 ppm
Test Value	15
SD	3.4
RSD	22%

n = 15
% of Rec. Value 60%

BCSS (NRC, Canada)

Rec. Value	18.5 ppm
Test Value	9.5
SD	2.4
RSD	25%

n = 16
% of Rec. Value 51%

Blank values were run after every 10 samples. All blank values were less than 1 ppm except 2, which were 1 ppm.

TABLE 6B. 1985 RESULTS OF WHOLE ROCK STANDARDS RUN IN MULTIELEMENT ICAP ANALYSIS (ppm).

	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	MnO	P_2O_5
NIM-S (syenite, South Africa)							
Rec. Value	17.34	1.40	0.68	0.46	15.35	0.01	0.12
Test Value (n = 4)	17.68	1.43	0.69	0.47	15.39	0.01	0.13
SD	0.08	0.03	0.06	0.01	0.18	0	0.01
NIM-L (lujavrite, South Africa)							
Rec. Value	13.64	9.91	3.22	0.28	5.51	0.77	0.06
Test Value (n = 2)	13.55	10.13	3.14	0.26	5.25	0.76	0.05
DR-N (diorite, France)							
Rec. Value	17.56	9.69	7.09	4.47	1.73	0.21	0.25
Test Value (n = 4)	17.81	9.47	6.88	4.21	1.64	0.21	0.23
SD	0.15	0.09	0.06	0.05	0.05	0	0.01
GS-N (granite, France)							
Rec. Value	14.73	3.76	2.51	2.31	4.64	0.06	0.28
Test Value (n = 2)	14.87	3.73	2.43	2.28	4.49	0.05	0.28
GA (granite, France)							
Rec. Value	14.51	2.77	2.45	0.95	4.03	0.09	0.12
Test Value (n = 4)	14.92	2.76	2.42	0.94	4.04	0.08	0.13
SD	0.20	0.04	0.05	0.02	0.04	0	0.01

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